### Fourth Quarterly Report

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HERMETICALLY-SEALED NICKEL-CADMIUM CELLS
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### STUDY OF PROCESS VARIABLES ASSOCIATED WITH MANUFACTURING HERMETICALLY-SEALED NICKEL-CADMIUM CELLS

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### FOURTH QUARTERLY REPORT

FOR

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### ABSTRACT

This report describes the fourth quarter of a program to determine and study the critical process variables associated with the manufacture of aerospace, hermetically-sealed, nickel-cadmium cells. The work consummated in the fourth phase was primarily concerned with the determination and study of the process variables associated with the positive and negative plaque impregnation/polarization process. The experimental data resulting from the implementation of fractional factorial design experiments are analyzed by means of a linear multiple regression analysis technique. This analysis permitted the selection of preferred levels for certain process variables to achieve desirable impregnated plaque characteristics. Assuming the maximization of plaque pick-up weight in conjunction with good electrical capacity and physical characteristics is desired, the following are some of the major conclusions drawn from the results of this effort:

### POSITIVE PROCESS VARIABLES

A high nickel nitrate solution specific gravity is preferred in the positive plaque impregnation/polarization process.

A high nickel nitrate solution temperature is preferred.

A long impregnation time is preferred.

A short polarization time is preferred.

### NEGATIVE PROCESS VARIABLES

A low cadmium nitrate solution specific gravity is preferred in the negative plaque impregnation/polarization process.

A long impregnation time is preferred.

A long polarization time is preferred.

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### I. INTRODUCTION

The objective of this program is to develop a process procedure and control for manufacturing nickel-cadmium aerospace cells with reliable five (5) year life capability. In order to achieve these objectives, each component part will be investigated separately and collectively to determine the critical variables and related interactions.

The total program consists of four (4) distinct, yet interrelated phases. The first phase consisted of a detailed analysis of our procedures in conjunction with a review of pertinent literature of nickel-cadmium batteries to assess critical variables of the various processes that affect cell performance. The second phase will involve the evaluation and testing (verification) of the variables and their interrelation as determined in Phase 1. This will include a design of experiments to experimentally identify critical variables and to establish tolerances required for uniform performance. Phase 3 includes the detailed preparation of a Quality and Reliability Assurance Program, Acceptance and Manufacturing Flow Sheets and a complete hardware specification that can be practically implemented in a cost effective manner. This specification will be patterned after "S-716-P-23, Interim Model Specification for High Reliability Nickel-Cadmium Spacecraft Cells." The Fourth Phase of the program will be to implement the results of Phases 1 through 3 on a production basis. This effort will "prove out" the conclusions and will establish both validity of concept and applicability to production equipment and overall operational capability. During this phase, the deliverable items of separation, positive and negative plates will be prepared. Also, 20 nickel-cadmium cells of 20 ampere-hour size will be manufactured to the developed procedure. Inspection levels will be 100% minimum and complete traceability maintained.

The first quarter of this program was devoted to investigating the dry-sintering process used in manufacturing porous nickel plaque.

A factorial experiment was designed to examine the sintered plaque character—
istics as a function of the process variables. The data gathered from this experiment were analyzed using a step-wise multiple regression technique designed for use with the IBM 1130 computer. At the completion of analysis, plaques with different characteristics were selected for use in the impregnation factorial experiment.

The second quarter's work encompassed additional variability analyses of the data from the unimpregnated plaque experiments. This effort included the prediction of the variability of the plaque manufacturing process within the normal tolerance range of the equipment and was instrumental in determining the critical variables and their effects upon the process for the purpose of instituting new control limits. This was followed by a short production run of three (3) different types of plaques (high strength-low porosity, average strength-average porosity and low strength-high porosity). These plaques are being used for the impregnation studies of the experimental program. Also during the second quarter, major effort was directed toward the setting up of a separate facility for the impregnation, polarization, formation, washing and other items related to the cell assembly for the contract. An experimental program was designed to study impregnation, polarization, formation and washing.

During the second reporting period, work began on the preparation of specifications for separator, ceramic-to-metal seal cover assembly and unimpregnated plaque manufacturing control.

The third quarterly period included an investigation (strength tests and scanning electron micrographic analysis) of the properties of the three groups of sintered plaques produced for use during the impregnation/polarization study. This effort revealed an anomalous group of low strength which was attributed to a high sintering temperature in conjunction with a strong sintering furnace reducing atmosphere. This investigation also revealed a plaque group exhibiting characteristic highly desirable in a nickel-cadmium cell plaque; this included high strength and a very uniform distribution of the porous void.

The experimental data resulting from previously performed impregnation/polarization design experiments was analyzed to determine the effects of certain process variables which were selected for investigation in the present process study. This information was used during the following period to reduce the number of process variables considered for study in the new impregnation/polarization design experiments.

Preliminary drafts of Quality Assurance Specifications for sintered nickel plaques, separator materials, and ceramic-to-metal seals were also completed during this period.

This fourth quarter period of the program was devoted to the implementation and evaluation of the impregnation/polarization factorial design experiments formulated for the study of the associated process variables. The first section of this report presents the experimental data and discusses problems encountered which required the reformulation of design experiments. The second section presents the results of a linear multiple regression analysis of the experimental data, and discusses the interpretation of these results. The last section presents the conclusions and recommendations which resulted from the total effort performed during the subject period.

### II. IMPREGNATION/POLARIZATION EXPERIMENTAL WORK

### A. <u>Impregnation/Polarization Process</u>

An impregnation/polarization process is the means by which the active material of the nickel-cadmium couple is impregnated in the porous sintered nickel plaque. In the Eagle-Picher process, impregnation is accomplished by immersion of the unimpregnated plaques in a hot, slightly acidic solution of either nickel or cadmium nitrate. This operation may be performed under a vacuum. Polarization or the conversion of the electrically inactive nitrate form of nickel or cadmium to the electrically active hydroxide form is accomplished by immersion of the impregnated plaques in a strong caustic solution (may be either sodium hydroxide or potassium hydroxide) accompanied by the passage of a polarizing cathodic current. The plaques are then washed in deionized water to remove the caustic solution.

The above operation is repeated until a predetermined amount of active material impregnation is achieved. The impregnated plaques are then subjected to an electrochemical cleaning or formation operation in preparation to their fabrication into cells.

### B. Impregnation/Polarization Study

To study the effects of the numerous process variables associated with the impregnation/polarization process, a statistical, partially replicated, fractional factorial design experiment was formulated to measure impregnated plaque characteristics as a function of the process variables. This technique permits the evaluation of a large number of variables with a limited amount of experimental work.

The design experiments for the positive plaque impregnation/
polarization study and the negative plaque impregnation/polarization
study were presented in Section III.B of the Second Quarterly Report.

(1)
For continuity, they will be presented at the beginning of the experimental data sections of this report.

A multiple regression program design for use with the IBM Model 1130 digital computer will be used for data processing during this work. The program evaluates the data resulting from the design experiments by weighing each variable with respect to its effect upon a particular impregnated plaque characteristic i.e., pickup weight; and then calculates a numerical value for its significance to the overall process. More detail will be given concerning the multiple regression analysis technique in the data analysis section of this report.

Pertinent information is contained in both the Second and Third Quarterly Reports<sup>(2)</sup> concerning the impregnation/polarization study. The following sections should be reviewed for a more thorough understanding of the present work.

Section III.C of the Second Quarterly Report contains a discussion and photographs of the scaled down production equipment used during the experimental phase of the present effort. A brief description of the impregnation/polarization tank, formation tank, nitrate/caustic storage tanks, and the deionized water and power supply systems may be found therein.

Section III.D of the Second Quarterly Report and Section III.B of the Third Quarterly Report describes the physical and chemical test procedures used during the program to gain information about the process and outlines a general implementation procedure to fulfill the intent of the subject study.

Section IV of the Third Quarterly Report contains a discussion of a standardized formation or electrochemical cleaning process which all of the impregnated plaques resulting from the impregnation/polarization study were subjected to.

One of the responses measured by the design experiment is termed plate capacity. Section V of the above Quarterly Report presents a description, by means of a discussion and a photograph, of the equipment and procedures utilized to measure the electrical capacity of a single positive or negative plate. All plate capacities listed in this report have been determined by the indicated standardized procedure.

A portion of the information contained in the above referenced sections is restated here for continuity in this report.

Three groups of unimpregnated plaques were produced for use during the impregnation/polarization study to assess the effects of raw plaque variation upon impregnated plaque characteristics. Lot 151 consists of plaques of high strength and low porosity, Lot 153 consists of plaques of medium strength and medium porosity, and Lot 152 consists of plaques of low strength and high porosity. Specific information concerning these three groups of plaques is contained in section II of the Third Quarterly Report.

As the impregnation/polarization tank is large enough to accommodate only ten plaques, a sample size had to be selected accordingly. Three plaques were selected from each of the three plaque lots described above; these nine plaques then constituted the sample for an experiment. This procedure offers several advantages; time was saved by the evaluation of three variables (three different types of plaques) with a single

experiment and the three types of plaques are assured of exposure to identical experimental conditions. The formation tank is larger than the impregnation/polarization tank and can accommodate 20 plaques. For maximum utilization, the procedure followed during this study was to complete two impregnation/polarization experiments and then subject the resulting 18 plaques to the formation operation.

### C. Positive Impregnation/Polarization Experiments

TABLE I
POSITIVE IMPREGNATION STUDY

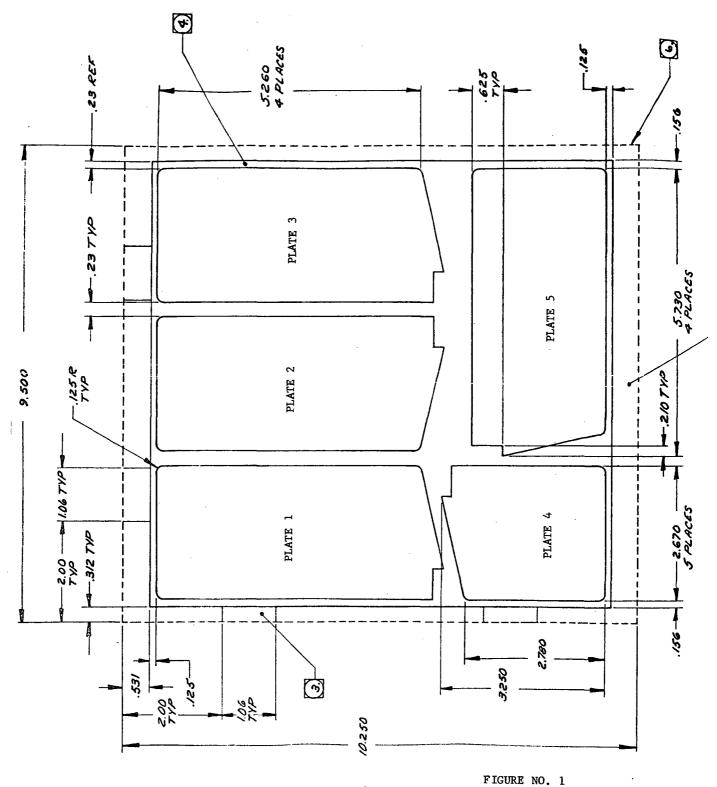
<u>Variables</u>	Designation	<u>Levels</u>
1	Specific Gravity of Nickel Nitrates	1.700 - 1.800
2	Free Acid	1 - 4 gm liter
3	Temperature of Nitrate	140°F - 200°F
4	Time of Impregnation	15 Minutes - 1 Hour
5	Vacuum	0 - 15 Inches
6	Wash Time	10 - 30 Minutes
7	Wash (Number of Cycles)	1 - 3
8	Wash Water Temperature	R.T 150°F
9	pH of Wash Water	Measured
10	Type of Caustic	KOH - NaOH
11	Specific Gravity of Caustic	1.200 - 1.300
12	Temperature of Caustic	80°F - 150°F
13	Amount of NH <sub>3</sub> in Caustic	Measured
14	Amount of $CO_2$ in Caustic	Measured
15	Amount of OH in Caustic	Measured
16	Polarization Current	.14 amps/sq.in.
17	Polarization Time	15 minutes - 1 hour
18	Voltage of Plaque of Ref. Electrode	Measured
19	Amount of Cycles with Same Caustic	1 - 5
20	Number of Total Cycles	Measured
21	Type of Plaque	3 Types
RESPONSES		
1	Pick-up Weight	
	Capacity	
2 3	Plaque (Characterization)	

The following interpretation may be applied to the three responses for both the positive and negative impregnation/polarization design experiments:

- Pickup Weight This is simply the amount of active material impregnation determined by subtracting the starting weight of the unimpregnated plaque from the weight of impregnated plaque after completion of the formation process.
- 2. Capacity This is the electrical capacity of a single plate expressed in ampere-hours and determined by a procedure previously described (see Section V of the Third Quarterly Report).

The capacity test procedure used in this program involved the selection of three plates from each group of three plaques, one plate from each plaque. A plaque contains five plates, a top row of three and a lower row of two (see Figure 1). For identification, the plates are assumed to be numbered as if reading a page in a book. The top three plates are numbered from left to right 1, 2, and 3; and the lower plates are 4 and 5.

Normally, the middle plate in the top row (Plate Number 2) is selected from the first plaque. From the second plaque, the second plate in the lower row (Plate Number 5) is usually chosen. Then, for the third plaque, Plate Number 2 is selected again. This procedure allows the evaluation of the difference in plate capacity from plaque to plaque within a group of assumed identical plaques and evaluation of the capacity difference associated with different areas



of a plaque.

Two terms are used to express electrical capacity: The first term is ampere-hours/plate which is the capacity of a single standard (approximately 15 in<sup>2</sup>) plate used through-out this work. The second term is ampere-hours/gram which may be considered an efficiency factor. The actual grams of active material impregnated in a test plate is determined and the ampere-hours capacity is expressed in terms of this quantity.

In addition to the three responses listed above, selected positive and negative plaques were subjected to an individual plate dimensional analysis before and after the impregnation/polarization process. The purpose of this analysis was to determine if there was any growth in plates associated with the operation. The dimensional data for the selected plaques may be found in Appendix B of this report.

### 3. Plaque Characterization

This is a subjective assessment of a plaque or group of plaques relative physical appearance or degree of cleanliness. For the purpose of entering data in the regression program, a particular plaque appearance is assigned a numerical value which rates it with respect to other plaque appearances.

### PLAQUE CHARACTERIZATION RATING SYSTEM

- 5.0 Best impregnated plaques produced, smooth clean surfaces, no cracks.
- 4.0 Good plaques, smooth clean surfaces, minor cracks.
- 3.0 Fair plaques, rough surfaces, moderate cracking.
- 2.0 Marginal plaques, very rough surfaces, severe cracking, mostly unsatisfactory for use.
- 1.0 Very poor plaques, blistered surfaces, material flaking off, completely unsatisfactory for use.

The following photographs (See Figures 2 and 3) demonstrate the physical appearance of two plaques which received a 1.0 rating. The first photograph is of Plaque No. 2, Lot 152, Experiment No. 9B after completion of the impregnation/polarization process. The second photograph is of plaque No. 2, Lot 152, Experiment No. 10B after completion of the formation process. In addition, a photograph (see Figure 4) included in the discussion section of this report demonstrates the physical appearance of two plates receiving 5.0 and 3.0 ratings.

In the descriptions of variable levels for a particular experiment, the actual measured values, where applicable, were reported.

Normally, an impregnation/polarization experiment involves three to four cycles; in the case where a variable's value changes between cycles, the maximum and minimum values are recorded.

Space did not permit the inclusion of all plaque to reference voltage measurements (variable Number 18) recorded during the process in the data section. This data may be found in the Appendix A of this report. Considerable difference will be observed in the voltage between similar experiments as the result of experimentation with different reference electrodes. The reference electrode used for some experiments was a standard nickel-cadmium negative plate, in others a positive plate was used, and in still others, the side of the impregnation/polarization tank was used as a reference electrode. In an interpretation of the plaque to reference voltages, these differences will have to be taken into consideration.

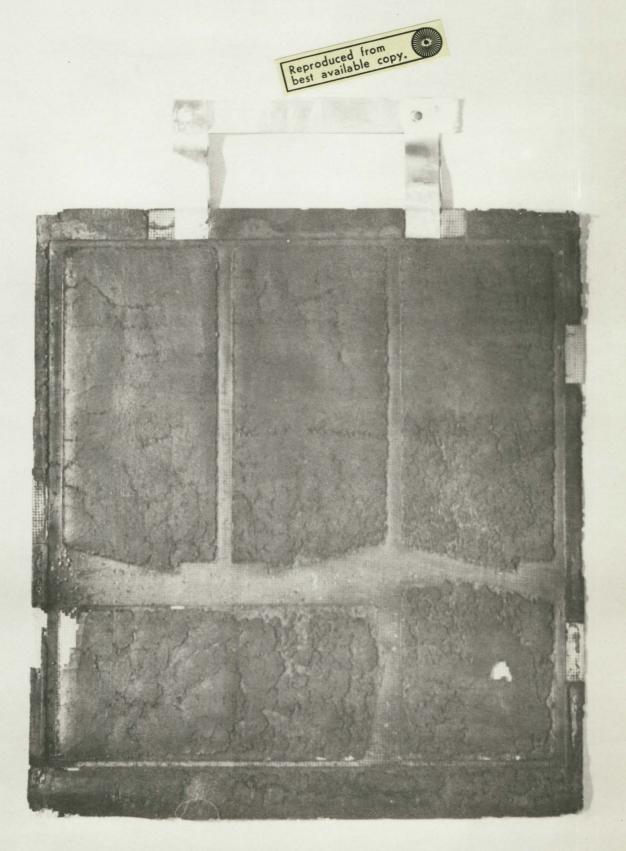
POSITIVE IMPREGNATED PLAQUE
AFTER COMPLETION OF THE
IMPREGNATION/POLARIZATION PROCESS

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FIGURE 2

POSITIVE IMPREGNATED PLAQUE AFTER COMPLETION OF THE FORMATION PROCESS



### D. Reassessment of Positive Experimental Work

During the initial implementation of the positive design, experiments, two problems arose. The first problem was associated with the physical integrity of the impregnated plaques. A number of the plaques exhibited a "blistering" phenomenon which resulted in the loss of the nickel matrix and the impregnated active material. It appeared the degree of latitude available in the selection of process variable levels was considerably more limited than originally assumed. The second problem involved the time element allocated to this phase of the study. A re-examination of the study time factor in light of the experience gained from the first four experiments indicated this phase of the overall program would consume considerably more time than allotted in the planned scope of work.

As a solution to the first problem, the data resulting from the first four experiments was reviewed in an attempt to determine which variable and its level or variables and their levels were most influential in affecting the poor results. It became obvious that Experiment Number Two produced somewhat better appearing plaques than the other experiments and that it differed in being impregnated under 0 inches of vacuum. It was decided to set this variable (Number 5) constant at its lower level (0 inches of vacuum) and continue the experiment.

As a solution to the second problem, it was necessary to reduce the original list of process variables (21) considered for investigation. The regression program requires that at least one more experiment be performed than the number of variables to be evaluated. So by reducing the number of variables, the number of experiments required

to be performed for evaluation is reduced.

In this program, a variable is eliminated by making it a constant value in the experiments. To this end, a reduction in the number of variables was effected by the utilization of the applicable variable level preference data described in Section III.A of the Third Quarterly Report, and through a process of engineering judgment to determine which of the remaining variables is important enough to remain a variable and which should be relegated to constants.

A variable is set constant at one of the levels defined in the original design experiment. This action will permit the use of the data generated by the experiments performed during this work at some further date if it is decided to continue the study of process variables with the original design experiments. The following summarizes the results of this variable reduction effort:

### VARIABLES

- Specific Gravity of Nickel Nitrates Selected to remain a variable.
- 2 Free Acid Selected to remain a variable.
- Temperature of Nitrate The effect of this variable on the subject responses was determined by previous design experiments referenced above. Throughout this work, the desired impregnated plaque parameters were high pick-up weight, good electrical capacity and a smooth, clean appearance. The above design experiments demonstrated a preference for a high nitrate solution temperature to achieve these results. This variable was set constant at its higher level of 200°F.
- 4 Time of Impregnation Selected to remain a variable.
- 5 Vacuum Set constant at its lower level of 0 inches for reasons previously stated.
- Wash Time This variable and the next two concerned with plaque washing were set constant at levels convenient to the performance of the experiments, but which would still result in an adequate degree of washing as judged by the present production procedures. This first variable was set constant at its lower level of 10 minutes.

- Wash (Number of Cycles) Set constant at higher level of 3 cycles, see Variable Number 6.
- 8 Wash Water Temperature Set constant at lower level of room temperature, see Variable Number 6.
- 9 pH of Wash Water Experience with the first experiments indicated the value of this variable changed very little and did not appear to be very important to the process. This measurement is to be continued, but the variable will not be considered for entry in the regression program.
- 10 Type of Caustic selected to remain a variable.
- 11 Specific Gravity of Caustic Set constant at higher level of 1.30 for reasons stated in Variable Number 3.
- 12 Temperature of Caustic Set constant at higher level of 150°F for reasons stated in Variable Number 3.
- 13 Amount of NH3 in Caustic Same as Variable Number 9.
- 14 Amount of CO3 in Caustic Same as Variable Number 9.
- 15 Amount of OH in Caustic Same as Variable Number 9.
- 16 Polarization Current Set constant at higher level of 0.4 amps/in<sup>2</sup> for reasons stated in Variable Number 3.
- 17 Polarization Time Select to remain a variable.
- 18 Voltage of Plaque to Reference Electrode Same as Variable Number 9.
- Amount of Cycles With Same Caustic The first experiment performed utilized fresh caustic for each cycle and there was no discernable difference in the measured responses for this experiment with respect to the experiments which were cycled in the same caustic. Based on this and the need for expediency, this variable was set constant at the higher level of 5 cycles.
- Number of Total Cycles This measured variable was set constant at 4 cycles, it was felt this number of cycles would result in approximately the same amount of active material pick-up as experienced in the present production operation; and would render the results of this experiment work more comparable to the present "state-of-the-art".
- 21 Type of Plaque Selected to remain a variable.

The culmination of the above total effort is the following abbreviated list of process variables now subject of an investigation by the multiple regression analysis technique:

	<u>VARIABLES</u>	LEVELS
1	Specific Gravity of Nitrate	1.70 - 1.80
2	Free Acid	1.0 - 4.0 gms/liter
4	Time of Impregnation	15 Minutes - 60 Minutes
10	Type of Caustic	KOH - NaOH
17	Polarization Time	15 Minutes - 60 Minutes
21	Type of Plaque	3 Types

For identification purposes, the experiment numbers used to identify experiments performed under the provisions of this new abbreviated design experiment will be designated with the letter "A".

After the completion of three (3) more experiments under the provisions of the new shorter design experiment, it became apparent the old problem of impregnated plaque blistering with the associated flaking of active material was not yet resolved. The second attempt to solve this problem took the same approach as described before. The data resulting from the now seven performed experiments was reviewed to determine if a certain variable level or variables might be associated with the experiments exhibiting the most destructive effect upon the plaques. This approach revealed that polarization in a caustic solution KOH was a much more destructive environment than polarization in a caustic solution of NaOH under similar process conditions. The use of a high free acid nitrate solution content also appeared to afford a much more destructive environment than the use of low free acid levels. The combination of the above two variable levels in one experiment resulted in the complete destructive

tion of all impregnated plaques associated with the experiment.

It was elected to relegate these two variables (Numbers 2 and 10) to constants at their levels more compatible to the impregnation/polarization process (1.0 gm/liter and NaOH) and to continue the study with the investigation of the remaining variables.

Positive experiments performed from this point are additionally designated with the letter "B".

### E. Negative Impregnation/Polarization Experiments

TABLE II

### NEGATIVE IMPREGNATION STUDY

<u>Variables</u>	Designation	Levels
1	Specific Gravity of Cadmium Nitrate	1.800 - 1.900
2	Free Acid	.25 gm/liter
3	Temperature of Nitrate	110°F - 140°F
4	Time of Impregnation	15 Minutes - 1 Hour
5	Vacuum	0 - 15 Inches
6	Wash Time	10 Minutes - 30 Minutes
7	Wash (Number of Cycles)	1 - 3
8	Wash Water Temperature	R.T 150°F
9	pH of Wash Water	Measured
10	Type of Caustic	KOH - NaOH
11	Specific Gravity of Caustic	1.200 - 1.300
12	Temperature of Caustic	80°F - 150°F
13	Amount of NH3 in Caustic	Measured
14	Amount of CO <sub>2</sub> in Caustic	Measured
15	Amount of OH in Caustic	Measured
16	Polarization Current	.14 Amps/sq.in.
17	Polarization Time	15 Minutes - 1 Hour
18	Voltage of Plaque to Ref. Electrode	Measured
19	Amount of Cycles with Same Caustic	1 - 5
20	Number of Total Cycles	Measured
21	Type of Plaque	3 Types

### Responses

- 1 Pick-up Weight
- 2 Capacity
- 3 Plaque (Characterization)

As in the case of the positive design experiment, the original negative design experiment was subjected to a variable reduction effort to derive a more feasible experimental program. The same variables finally selected for investigation during the positive impregnation/polarization study were also chosen for the negative study.

Again, utilizing the impregnation/polarization multiple regression data presented in Section III.A of the Third Quarterly Report,

the applicable variables of the negative design experiments were set constant at the indicated preferred levels. The following summarizes the subject variables and the preferred levels:

### VAR IABLES

- 3 Temperature of Nitrate High nitrate temperature was preferred in the previous design experiments. This variable was set constant at the higher level of 140°F.
- 11 Specific Gravity of Caustic High level preferred, set constant at higher level of 1.30.
- 12 Temperature of Caustic Low level preferred, set constant at lower level of  $80^{\circ}F$ .
- Polarization Current High level preferred, set constant at a higher level of 0.4 Amps/in<sup>2</sup>.

The remaining variables relegated to the status of constants, and the measured variables are treated in a fashion identical to the positive experimental work. The higher free acid level of variable Number 2 was chosen because this is very close to the level presently used in the production operation (0.4 grams/liter free acid).

The following summarizes the negative impregnation/polarization process variables selected for study in the revised negative design experiment:

	<u>VAR IABLES</u>	LEVELS
1	Specific Gravity of Nitrate	1.80 - 1.90
4	Time of Impregnation	15 Minutes - 60 Minutes
17	Polarization Time	15 Minutes - 60 Minutes
21	Type of Plaque	3 Types

### F. Positive and Negative Impregnation/Polarization Experimental Data Table Number III is a composite of the process variable values recorded throughout the impregnation/polarization process study.

Table Number IV is a composite of the response values recorded throughout the process study. Except for the plaque characterization rating (which is the same for all three plaques which make-up a single experiment), the individual response values are an average of three measurements for each response.

Individual measurements for each response and specific comments concerning the plaque characterization response may be found in Appendix C of this report.

TABLE NUMBER III

POSITIVE IMPREGNATION/POLARIZATION EXPERIMENT VARIABLE LEVELS

VARIABLES  1. Sp.Gr. of Nitrate	1	,	·	,	l					
1. Sp.Gr. of Nitrate		7	7	4	5 <b>A</b>	<b>V</b> 9	7.A	88	- <b>V</b> 6	10B
	1.70	1.70	1.70	1,70	1.70	08.1	1.80	1.80	1.80	1,70
Free Acid (Controlled 2. By Addition of HNC.)	gms/liter 1.0	1.2-1.4	1.0	1.0	0.8-1.0	3.8	3.4-4.4		1.0-1.2	1.0-1.8
Temp. of Nitrate (In I/P Tank) °F	132-137	136-145	175-189	180-188	178-186	182-190	188-194		186-190	182-195
4. Time of Impregnation	1 Hr.	15 Min.	15 Min.	1 Hr.	1 Hr.	1 Hr.	15 Min.	15 Min.	1 Hr.	15 Min.
5. Vacuum (Inches)	15	0	15	15	0	0	0	0	0	0
6. Wash Time (Minutes)	30	10	10	10	10	10	10	10	10	10
7. Wash (No. of Cycles)	1	1	3	3	3	3	3	3	Э	3
N N 8. Wash Water Temp. (°F)	59	64-70	52-54	50-56	50-52	48-50	46-48	48-49	49	48
pH of Wash Water 9. (Measured pH Pape	4.5-5.3	4.5-5.0	4.5-5.3	4.5	4.5	4.5	4.5	4.5	4.5	4.0
10. Type of Caustic	NaOH	КОН	КОН	КОН	NaOH	NaOH	КОН	NaOH	NaOH	NaOH
11. Sp.Gr. of Caustic	1.30	1.20	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30
12. Temp. of Caustic (I/P Tank)	74-19	140-151	150-158	150-156	150-160	150-160	150-160	150-170	138-156	135-148
1	0.021- 0.037	0.008 <del>-</del> 0.013	0.010- 0.015	0.010- 0.016	0,007- 0,018	0.009- 0.024	900*0	0.014- 0.026	0.012- 0.014	0.009- 0.010
Amt. of CO <sub>3</sub> 14. in Caustic (N)	0.2	0.24- 0.44	0.84-* 1.40	0.44- 0.60	0.28- 0.44	0.16- 0.24	0.12	0.20- 0.44	0.32- 0.36	0.32
15. Amt. of OH in Caustic (N)	9.2	0.50- 0.55	7.1-7.4	7.0-7.4	8.6-7.6	9.6-9.6	9.7	9.2-9.7	9.2-9.5	8.9
16. Polarization Current	0.4 amps/in <sup>2</sup>	0.4	0.4	0.4	0.4	7.0	7.0	0.4	0.4	0.4
Polarization Time	1 Hr.	1 Hr.	15 Min.	1 Hr.	1 Hr.	1 Hr.	15 Min.	1 Hr.	15 Min.	15 Min.
18. Voltage of Plaque to Ref.	See Appendix	endix		:			1			1
19. Amt. or Cycles W/Same Caustic	1	4	4	4	4	4	4	4	. 7	7
20. No. of Total Cycles	7	7	4	4	4	7	7	4	4	4
21. Type of Plaque (Type)	3	3	3	3	3	3	3	3 3 3	3	3

TABLE NUMBER IIIA

NEGATIVE IMPREGNATION/POLARIZATION EXPERIMENT VARIABLE LEVELS

1. Sp. Gr. of Nitrate Free Acid (Controlled 2. By Addition of NHO3 3. Temp. of Nitrate (In I/P Tank) °F 4. Time of Impregnation 5. Vacuum (Inches) 6. Wash Time (Minutes) 7. Wash (No. of Cycles) 8. Wash Water Temp. (°F) 9. pH of Wash Water (Measured pH Paper) 10. Type of Caustic 11. Sp. Gr. of Caustic 12. Temp. of Caustic (I/P Tank) 13. Amt. of NH; in Caustic (I/P Tank) 14. Amt. of CO3 in Caustic (I/P Tank) 15. Amt. of OH in Caustic (N			EX PER IMENTS			
1. S 2. B 3. 3. 3. 6. 6. 6. 10. 11. 12. 11. 12. 11. 12. 11. 12. 11. 11	VAR IABLES	1	2	3	7	
2. B 3. B 3. B 5. 6 6. 7 7. 7 10. 11. 12. 13. 13. 14.	trate	1.90	1,90	1.80	1,80	
3. 5. 6. 6. 6. 6. 10. 10. 11. 12. 13. 13. 14. 14.	ntrolled of NHO3	gms/liter 0.4-0.5	0.5-0.6	0.4-0.6	0.5	
4. 5. 7. 7. 10. 11. 12. 13. 14.	rate (In I/P	130-138	132-140	130-138	128-135	
5. 6. 7. 7. 10. 11. 12. 13.	egnation	15 Minutes	1 Hour	1 Hour	15 Minutes	
6. 7. 8. 9. 10. 11. 12. 13.	les)	0 Inches	0 Inches	0 Inches	0 Inches	
7. 8. 9. 10. 11. 12. 13. 14.	(inutes)	10 Minutes	10 Minutes	10 Minutes	10 Minutes	
8. 9. 10. 11. 12. 13. 14.	Cycles)	3 Cycles	3 Cycles	3 Cycles	3 Cycles	
9. 10. 11. 12. 13. 14.	emp. (°F)	48 to 52	50	84	52 to <b>5</b> 4	
10. 11. 12. 13. 14.	<i>l</i> ater					
10. 11. 12. 13. 14.	Paper)	4.5	4.5	4.5	4.5	
11. 12. 13. 14.	tic	NaOH	NaOH	NaOH	NaOH	
1 1 1 1	austic	1.30	1.30	1.30	1.30	
111	ıstic				:	
		78-88	78–94	92-103	92-110	
1 1	Amt. of NH <sub>2</sub> in Caustic (N)	0.012-0.017	0.007	600.0	600*0	
l	in Caustic (N)	0.20-0.28	0.16	0.20-0.92*	0.16-0.20	
	Amt. of OH in Caustic (N)	8.6 to 9.1	8.3	9.0 to 9.3	7.8 to 8.7	
16. Polarization Current	Current	0.4 amps/in <sup>2</sup>	7.0	0.4	0.4	
17. Polarization Time	1 Time	1 Hour	. 15 Minutes	1 Hour	15 Minutes	
18. Voltage of P	Voltage of Plaque to Ref.		See Appendix			
19. Amt. of Cycl	Amt. of Cycles W/Same Caustic	3	3	3	3	
20. No. of Total Cycles	Cycles	3	3	3	3	
21. Type of Plaque (Type)	ue (Type)	3	3	3	3	

 $^*$ Suspect high carbonate indication is the result of the use of carbonate contaminated delonized water.

TABLE NUMBER IV
POSITIVE IMPREGNATION/POLARIZATION RESPONSE DATA (AVERAGED)

	EXPER IMENTS		-			2			3			7	
RESPONSES	PLAQUE LOTS	151	152	152 153 151 152	151	152	153	151	152	153	153 151 152 153 151 152	152	153
Pick-up Wt. (After Formation, Grams)		57.7	42.8	51.7	41.0	43.3	2.8 51.7 41.0 43.3 41.7 46.0	0.91	*	*	*	*	*
Ampere-Hours/Plate		2.41	1.91	1.91 2.05 1.41 *	1.41	*	1,38		*	*	*	*	*
Ampere-Hours/Gram		0.227	0.220	0.220 0.288 0.217	0.217	*	0.222	0.222 0.234 *	*	*	*	*	*
Characterization Rating		3.0	2.0	2.0	3.0	1.0	2.0 2.0 3.0 1.0 2.0 2.0 1.0 1.0 1.0 1.0	2.0	1.0	1.0	1.0	1.0	1.0

	153		. 63	.231		
	i	* 45.0	1.(	0.2	3°(	
10B	152		*	*	1.0	
	151	8*95	1.93	*	0.0 5.0 1.0	
	153	67.8 46.8	*	0.249	1.0	• •
9B	152	*	*	*	1.0	se valu
	151	66.5	2.91	*	4.0	respons
	153	56.3	2.41 2.25 2.20 2.91	0.243	1.0 4.0 4.0 1.0	on of a (AVERAG
. 8B	152	51.7	2.25	0.261 0.247 0.243	1.0	rminatio SE DATA
	151	56.5 51.7 56.3 66.5	2.41	0.261	5.0	*The poor physical condition of the impregnated plaques prevented the determination of a response value. NECATIVE IMPREGNATION/POLARIZATION RESPONSE DATA (AVERAGED)
	153	*	*	*	1.0	evented AR IZATI
7.A	152	*	*	*	1.0	ques pr ION/POL
	151	*	*	*	1.0	ted pla
	153	61.7	3.01	0.282	0.4	ne impregna NEGATIVE IN
6A	152	*	*	*	1.0	of the
	151	60.2	3.08	0.294	5.0	dition o
	153	57.5	2,41	0.242	2.0	cal con
5A	152	55.8 62.8 57.5	3.05	0.257	1.0	or physi
	151	55.8	2.44	0.265	5.0	*The po

	EXPER IMENTS		-			2			3			7	
RES PONSES	PLAQUE LOTS	151	152 153 151 152 153 151 152 153 151 152 153	153	151	152	153	151	152	153	151	152	153
Pick-up Weight (After Form., Gms)		. 46.3	53.9 47.9 63.1 73.9 63.2 66.2 69.8 54.7 47.2 60.5 43.0	47.9	63.1	73.9	63.2	66.2	8.69	54.7	47.2	60.5	43.0
Ampere-Hours/Plate	-	1.97	2.33 2.05 2.63 3.10 2.80 3.04 3.29 2.31 2.30 2.82 2.00	2.05	2.63	3.10	2.80	3.04	3.29	2.31	2.30	2.82	2.00
Ampere-Hours/Gram		0.270	0.253 0.265 0.247 0.233 0.257 0.258 0.272 0.270 0.275 0.278 0.293	0.265	0.247	0.233	0.257	0.258	0.272	0.270	0.275	0.278	0.293
Characterization Rating		4.0	3.0 4.0 5.0 4.0 5.0 4.0 1.0 5.0 5.0 4.0 4.0	4.0	5.0	4.0	5.0	4.0	1.0	5.0	5.0	4.0	4.0

### III. DATA ANALYSIS

The analysis of the results from the impregnation/polarization design experiments described in the previous section was performed by means of a coded, stepwise linear multiple regression and correlation program designed for use with the IBM Model 1130 digital computer. (3)

A linear regression program assumes that each dependent variable (response) is a linear function of each of the independent variables. Through a multiple correlation coefficient technique, the program combines the effects of all the relationships among the dependent and independent variables and yields a single numerical measure of the overall relationship in the form of a regression equation.

$$Y = C + B_1 X_1 + B_2 X_2 + ... + Bn Xn$$

In the above example, the Y terms represents a dependent variable (response); the  $X_1, X_2, \ldots, X_n$  terms represent independent variables (values of the process variables); C is a constant term; and the  $B_1, B_2, \ldots, B_n$  terms are the respective equation coefficients. Through the interpretation of these coefficients, the effect of each independent variable upon a particular dependent variable may be determined.

The raw data consisting of values of independent variables is coded by the regression program prior to its compilation in the regression matrix for further processing. This operation standardizes the numerical values of each independent variable which are usually of different magnitudes. The maximum value of each independent variable is set equal to +1 and the minimum value is set equal to -1. All intermediate values are prorated between these two values. The use of coded raw data permits the direct comparison of the coefficients within a regression. The independent variable associated with the largest coefficient exerts the greatest effect upon the

subject response.

The following computer print-outs summarizes the regression analyses performed on the impregnation/polarization design experiment data. There are four major regressions (Tables I, II, III and IV); each subdivided into separate regression for each response (Tables Ia, Ib, etc.).

POSITIVE DESIGN EXPERIMENT, REGRESSION I

RAW DATA LISTING, LOT 151

	8 PLAQUE CHARACTERI- ZATION ALL LOTS AVERAGED	3.70	3.70	3.70	3.00	3.00	3.00	2.30	2.30	2.30	2.00	2.00	2.00
RESPONSES	7 PLAQUE PI CHARACTERI- ZATION LOT 151	ა იე •	5.00	5.00	00.4	00.4	00.4	5.00	5.00	5.00	3.00	3.00	3• ∪ ∪
	6 GRAM CAPACITY - (AMP-HRS)	0.26	0.25	0.27	0.25	0.25	0.26	0.27	0.27	0.27	0.25	0.25	0.24
	5 PLATE CAPACITY - (AMP-HRS)	2.37	2.59	2.37	2.41	2.47	2.36	2.82	2.97	2.94	1.93	2.02	1.85
	PICKUP WEIGHT - (GRAMS)	54.00	57.50	26.00	26.00	5.7.00	56.50	67.00	65.50	67.00	46.50	46.50	47.50
VARIABLES (X)	3 POLAR IZATION TIME - (MINUTES)	00•09	00.09	00•09	00.09	00.09	00.09	15.00	15.00	15.00	15.00	15.00	15.00
	2 IMPREGNATION TIME - (MINUTES)	00.09	00.09	60.09	15.00	15.00	15.00	00.09	00.09	. 00.09	15.00	15.00	12
Λ	1 SPEC IF IC GRAV ITY OF NITRATE	1000			80			Ο α: •			1,470		S N
	SPEC GRAN OI NITE	• 02											•

CORRELATION MATRIX

00	-0.152	0.380	0.912	0.114	0.177	0.256	0.573	1.000
7	0.301-	0.904	0.301	0.820	0.849	0.771	1.000	0.573
9	0.459	0.673	0.076	0.764	0.734	1.000	0.771	0.256
'n	699.0	0.712	600.0	0.975	1.000	0.734	0.849	0.177
4	1.000 0.000 0.000 0.724 0.669 0.459 0.301-0.152	0.000 1.000 0.000 0.677 0.712 0.673 0.904 0.380	0.000 0.000 1.000-0.035 0.009 0.076 0.301 0.912	0.724 0.677-0.035 1.000 0.975 0.764 0.820 0.114	0.669 0.712 0.009 0.975 1.000 0.734 0.849 0.177	0.459 0.673 0.076 0.764 0.734 1.000 0.771 0.256	0.301 0.904 0.301 0.820 0.849 0.771 1.000 0.573	0.114
m	00000	00000	1.000-	•0•035	600.0	0.076	0.301	0.912
2	00000	1.000	00000	0.677	0.712	0.673	0.904	0.380
	1.000	00000	000.0	0.724	699.0	0.459	0.301	-0.152 0.380 0.912 0.114 0.177 0.256 0.573 1.000
	-	~	m	4	Ŋ	\$	~	œ

## REGRESSION IA

# RESPONSE (Y), PICKUP WEIGHT

					RESIDUALS SSQS 3-3610925 6-1388874 6-16666874 6-666660 6-666660 8-166660 8-2777729 8-3888798 8-3888798
-					
OF ≻					Σ14000000000 000000000000000000000000000
S I S					αι ι Ι Ι Ι ο z
ANALYS	ESIDUAL 1.10417				00000000000000000000000000000000000000
156 T 156 T 1565 100 882	8				· σ
~mmm	RSQR 0.985				00000000000000000000000000000000000000
0000 mwww	0F2 8				w
	0F1	<b>-</b> 4		SNO	<ul><li></li></ul>
5.00 EFF	MULT F 175.57	, , ,	41666	EDICTIO	> \unu\unu\unu\unu\unu\unu\unu\unu\unu\un
X000 X000 X000	NSTANT •41666	LES IN MOD 50087 50830 602497	T= 56.	LS AND PR	> www.ww.wo.o.o.q.q.q.q.q.q.q.v.o.v.o.v.w.v.w.v.o.v.o.v.o.w.w.w.v.o.o.o.o
XHUW KOOO	000	>	CONSTAN	RESIDUAL	0 E 1114 N40m4m4r4b6010

## REGRESSION Ib

# RESPONSE (Y), PLATE CAPACITY

~		RESIDUALS SSUS CON CONTRACTOR CON
YSIS OF Y 33317E-02 02		
ANAL 64 12 12 8175017E-		<ul> <li>NUNDANDANDANDANDANDANDANDANDANDANDANDANDAN</li></ul>
SE (B) 0.0260 0.0260 0.0260 0.0260 0.0260 0.0260		Aununanaoouna Turanananaoouna Turanananaoounan Turanananaoounan
B COEF 0 • 2366 0 • 05316 0 • 0533 MULT F DF1 58 • 40	FL - Y 2 42499	> > C
000 000 000 000 000 000 000 000 000 00	ABLES IN MOD 0.2366 0.2366 0.02516 0.0033 TANT= 2.	> NUNUNUNUNUNUNUNUNUNUNUNUNUNUNUNUNUNUNU
メもろを	>	0 <b>0</b>

### REGRESSION IC

## RESPONSE (Y), GRAM CAPACITY

8			RESIDUALS SSQS 0.000000000000000000000000000000000
YSIS 99975E102 33296E102 33287E103 04			N
7 ANAL 0.5850007E			00000000000000000000000000000000000000
SE(B) 0.0022 0.0022 0.0022 0F2 RSQR DF2 RSQR			T I I I I I I I I I I I I I I I I I I I
B COEF 0.00049 0.00073 MULT F DF1 5.43	FL - Y 3 26216	EDICTIONS	> - 00000000000000000000000000000000000
RSGR X 0.00000 0.00000 0.0000 0.STANT	S IN MODE 0 00049 0 00078 0 00078 0 0008 0 0009	IDUALS AND PRE	> 00000000000 C • • • • • • • • • • • • • • • • • • •
<b>!</b> U	VARIABLE X 1 2 3 CONSTANT	RESID	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0

### REGRESSION Id

RESPONSE (Y), PLAQUE CHARACTERIZATION

	7					RESIDUALS S
Z.	YSIS OF Y	_ •06				>
CHARACIERIZAIION	1 3.99 2.00 3.99	RESIDUAL 0.7152558E-				00000000000000000000000000000000000000
(I), FLAQUE LOT 19	SE(B) 0.0002 0.0002 0.0002 0.0002	DF2 RSQR 8 0.999				m MO000000000000000000000000000000000000
KESYONDE	B COEF 0.2499 0.2499 0.2499	MULT F DF1	)EL - Y 4	25000	EDICTIONS	> mwmwwwwwwww #00000000000000000000000000000
	x000 0000 x000 x000 x000	CONSTANT 4.250003844	ABLES IN MOD 0.2499 0.7499 0.2499	1ANT= 4	IDUALS AND PR	>- mmm444mmmwww 0 • • • • • • • • • • • • • • • • • • •
	XHNM		> A &X40@	SNCD	RESI	0 8 1111 8 1100870010

### REGRESSION IE

RESPONSE (Y), PLAQUE CHARACTERIZATION ALL LOTS AVERAGED

			00000000000000000000000000000000000000
ر د			ж ш
YSIS OF Y 99949E-01 06			Σ 2000000000000000000000000000000000000
ANAL 10.99 ESIDUAL 92093EL			>0000000000000000000000000000000000000
SE(B) 0.0000 -1003.30 0.0000 2508.27 0.0000 6019.86 DF2 RSQR -0.11			n Noooooooooo Toooooooooo Tooooooooooooo
COEF • • • • • • • • • • • • • • • • • • •	L - Y 5 4999	EDICTIONS	~ www.www.www. ~ www.www.www. ~ www.www.www. ~ www.www.www. ~ www.www.www. ~ www.www.www.www. ~ www.www.www.www.www. ~ www.www.www.www.www.www. ~ www.www.www.www.www.www.www.www.www. ~ www.www.www.www.www.www.www.www.www.ww
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x40m	VARIABLE S	RESIDUALS	0 E HHH 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8

						· · ·	
-	VAK LABLES	(¥)	~	u	RESPONSES (	(Y)	c
SP.GR.	IMPREGNATION	POLAR IZATION	POROS ITY	PICKUP	PLATE	GRAM	PLAQUE
OF NITR <b>A</b> TE	TIME (MINUTES)	TIME (MINUTES)	(% VOID PLAQUE)	WEIGHT (GRAMS)	CAPACITY (AMP-HRS)	CAPACITY (AMP-HRS)	CHARACTERI- ZATION INDEX
1.70	00•09	00•09	83.30	54.00	2.37	0.27	
1.70	00.09	00.09	83.30	57.50	2.59	0.25	9.00
1.70	00.09	00.09	83.30	56.00	2.37	0.27	5.00
1.70	00.09	00.09	86.00	66.50	3.23	0.24	1.00
1.70	00.09	00.09	86.00	65.50	2.95	0.26	1.00
1.70	00.09	00.09	86.00	56.50	2.97	0.26	1.00
10°	00.09	00.09	85.70	54.00	2.16	0.25	5.00
1.70	00•09	00.09	85.70	29.50	2.54	0.22	5.00
1.70	00.09	00.09	85.70	29.00	2.54	0.24	2.00
11.80	15.00	00.09	83.30	26.00	2.41	0.25	4.00
12.80	15.00	00.09	83.30	57.00	2.47	0.25	4.00
13.80	15.00	60.09	83.30	56.50	2.36	0.26	4.00
1,1.80	15.00	00.09	86.00	48.50	2.05	0.25	1.00
1,80	15.00	00.09	86.00	52.00	2.28	0.23	1.00
1.80 16	15.00	00.09	86.00	54.50	2.43	0.25	1.00
17.80	15.00	00.09	85.70	56.50	2.17	0.23	4.00
1.80	15.00	00°09	85.70	57.00	2.32	0.24	4.00
19.80	15.00	00.09	85.70	55.50	2.12	0.25	4.00
1.80	60.09	15.00	83.30	67.00	2.82	0.27	2.00
7.1.40 2.1.40	60.09	15.00	83.30	65.50	2.97	0.27	2.00
1.80	60.00	15.00	83.30	67.00	2.94	0.27	5.00
1.70	15.00	15.00	83.30	46.50	1.93	0.25	3.00
1.70	15.00	15.00	83.30	46.50	2.02	0.25	3.00
1.70	15.00	15.00	83.30	47.50	1.85	0.24	3.00
1.70	15.00	15.00	85.70	45.50	1.70	0.23	2.00
1.70	15.00	15.00	85.70	46.00	1.65	0.22	2.00
27 1 0 70	15.00	15.00	85.70	43.50	1 53	£0.0	2.00

## CORRELATION MATRIX

	~	2	1 2 3 4	4	5	2 9	7	∞
-		0.350	1.000-0.350 0.158-0.088 0.302 0.175 0.246 0.095	088	0.302	0.175	0.246	960.0
2		1.000	-0.350 1.000 0.158-0.088 0.685 0.716 0.451 0.381	0.88	0.685	0.716	0.451	0.381
W	0.158	0.158	0.158 0.158 1.000 0.337 0.276 0.336 0.039 0.000	337	0.276	0.336	0.039	00000
4	-0.088-	0.088	-0.088-0.088 0.337 1.000-0.107-0.091-0.583-0.576	-00C	-701.07-	-0.091-	-0.583-	-0.576
r	0.302	0.685	0.302 0.685 0.276-0.107 1.000 0.924 0.526 0.335	101	1.000	0.924	0.526	0.335
ç	0.175	0.716	0.175 0.716 0.336-0.391 0.924 1.000 0.543 0.138	160	0.924	1.000	0.543	0.138
7	0.246	0.451	0.246 0.451 0.039-0.583 3.526 3.543 1.000 0.342	583	0.526	0.543	1.000	0.342
σc	0.095	0.381	8 0.095 0.381 0.000-0.576 0.335 0.138 0.342 1.000	576	3.335	0.138	0.342	1.000

### REGRESSION IIa

## RESPONSE (Y), PICKUP WEIGHT

			RESIDUALS
ALYSIS OF Y AL 39			
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### REGRESSION IIb

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### REGRESSION IIC

## RESPONSE (Y), GRAM CAPACITY

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## REGRESSION IId

# RESPONSE (Y), PLAQUE CHARACTERIZATION

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# NEGATIVE DESIGN EXPERIMENT, REGRESSION III

RAW DATA LISTING, LOT 151

	VARIABLES (X)				RES PONSES (	_	
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SPECIFIC	IMPREGNATION	IMPREGNATION POLARIZATION	PICKUP	PLATE	GRAM	PLAQUE	PLAQUE CHARACTER
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OF		•	1	ı	•	ZATION	ALL LOTS
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*FILES(4,FFFF2)	2)						
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0.0 • 4	15.00	00.09	43.90	1.84	0.26	4.00	3.70
06 • 40	00.09	15.00	54.10	2.71	0.24	5.00	4.70
1.90	00.09	15.00	63.00	2.63	0.24	5.00	04.70
1.90	00•09	15.00	62.20	2.56	0.24	5.00	4.70
	00.09	00.09	65.30	3.07	0.25	4 • 00	3.30
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11.80	15.00	15.00	38.00	1.61	0.25	5.00	4.30
12.80	15.00	15.00	52.00	2.65	0.27	5. €	4.30
1.80	1,315.00	15.00	51.50	2.63	0.28	5.00	4.30

### REGRESSION III



## CORRELATION WATRIX

1 2 3 4 5 6 7 8
1 1.000 0.000 0.000-0.101-0.391-0.329-0.000 0.371
2 0.000 1.000 0.000 0.928 0.749-0.777-0.000 0.371
3 0.000 0.000 1.000 0.957 0.040 0.131-1.000-0.928
4 -0.101 0.928 0.057 1.000 0.922-0.531-0.057-0.090
5 -0.391 0.749 0.040 0.922 1.000-0.215-0.040-0.183
6 -0.329-0.777 0.131-0.531-0.215 1.000-0.131-0.244
7 -0.000-0.000-1.000-0.057-0.040-0.131 i.000 0.928
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## REGRESSION IIIa

## RESPONSE (Y), PICKUP WEIGHT

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## REGRESSION IIID

## RESPONSE (Y), PLATE CAPACITY

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## REGRESSION IIIC

## RESPONSE (Y), GRAM CAPACITY

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## REGRESSION IIId

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### REGRESSION IIIe

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NEGATIVE DESIGN EXPERIMENT, REGRESSION IV

RAW DATA LISTING, LOTS 151, 152 AND 153

	∞	PLAQUE	CHAKACIEKI_		4.00	4.00	4.00	3.00	3.00	3.00	4.00	00•4	4.00	5.00	5.00	5.00	00.4	4.00	4.00	5.00	5.00	5.00	00.4
	7 GRAM	CAPACITY	(AMP-HRS)		0.26	0.27	0.26	0.26	0.25	0.24	0.27	0.26	0.25	0.24	0.24	0.24	.O.24	0.23	0.22	0.26	0.24	0.26	0.25
RESPONSES (Y)	6 PLATE	CAPACITY	(AMP-HRS)		1.98	2.08	1.84	2.42	2.39	2.17	2.12	2.15	1.89	2.71	2.63	2.56	3,00	3.12	3.18	2.64	2.90	2.85	3.07
	5 PICKUP	WE IGHT	(GRAMS)	1	47.20	47.80	43.90	54.90	55.30	51.40	48.00	49.10	76.60	64.10	63.00	62.20	73.00	75.10	73.20	62.00	63.00	63.90	65.00
•	4 POROSITY	(PERCENT	PLAQUE)	(	83•30	83.30	83.30	86.00	86.00	86.00	85.70	85.70	85.70	83.30	83.30	83.30	86.00	86.00	86.30	85.70	85.70	85.70	83.8
(x)	3 POLARIZATION	TIME -	(MINUTES)		00•09	00.09	00.09	00.09	00.09	00.09	00.09	00•09	00.09	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	00.09
VARIABLES (X)	2 IMPREGNATION	TIME -	(MINUTES)	( ( 1	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	00.09	00.09	00.09	00.09	00.09	00.09	00.09	00.09	00.09	00.09
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NEGATIVE DESIGN EXPERIMENT, REGRESSION IV (CONTINUED)

RAW DATA LISTING, LOTS 151, 152 AND 153

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0.26	0.25	0.26	0.25	0.25	0.25	0.28	0.27	0.25	0.27	0.28	0.27	0.27	0.27	0.29	0.27	Ú• 30
3.04	3.01	3.48	3.09	3.29	1.98	2.37	2.57	1.61	2.65	2.63	2.93	2.92	2.62	2.30	1.84	1 . 85
05.99	67.00	70.50	70.00	00*69	00.64	53.50	61.50	38.00	52.00	51.50	63.00	62.00	56.50	20.00	39.00	39.50
83.30	83.30	86.00	86.00	86.00	85.70	85.70	85.70	83.30	83.30	83.30	86.00	86.00	86.00	85.70	85.70	85.70
00.09	00.09	00.09	00.09	00.09	00.09	00.09	00.09	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
00.09	00.09	00.09	00.09	00.09	00.09	00.09	00•09	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	36
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### REGRESS ION IV

## CORRELATION MATRIX

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'n	5 0.055 0.754-0.095 0.171 1.000 0.943-0.560-0.269	0.754-	960.0-	0.171	1.000	0.943-	-0-560-	o
9	6 -0.152 0.645-0.116 0.151 0.943 1.000-0.356-0.347	0.645	-0.116	0.151	0.943	1.000-	-0.356-	0
7	7 -0.545-0.526-0.021 0.001-0.560-0.356 1.000 0.047	-0.526-	-0.021	0.001	-0.560	-0.356	1.000	်
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REGRESSION IVA

RESPONSE (Y), PICKUP WEIGHT

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### REGRESSION IV

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REGRESSION IV

RESPONSE (Y), PLATE CAPACITY

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The regressions contained in Tables I and III analyses data associated with the positive and negative design experiments utilizing responses which characterizes only Lot 151 plaques. Lot 151 plaques were elevated to this somewhat privileged position because they withstood the rigors of the experimental process better than the other two lots, and a greater degree of confidence is attached to the experimental results associated with them. The remaining regression, Tables II and IV, analyses the total data associated with the positive and negative design experiments respectively. The responses of all three lots of plaques are introduced into the regression picture by the incorporation of the plaque lot discriminating independent variables of plaque porosity.

A second plaque characterization response is associated with the regressions I and III. In this response, the numerical rating value is an average of the rating values for each lot of plaques in the experiment. The purpose of this additional response is to gain some insight into the harshness of the experimental conditions. For example, in a particular experiment, the stronger plaques of Lot 151 might exhibit a good physical appearance and be rated 5.0 while the remaining weaker two lots are very poor and receive ratings of 2.0 or 1.0. In a second experiment, Lot 151 plaques may again be very good and receive a rating of 5.0, but in this case, the two weaker lots are also good and receive ratings of 5.0 or 4.0. The average rating value for the second experiment would be considerably higher than for the first and permit the regression program to distinguish between the two experiments in reference to the harshness of the experimental conditions.

Referring back to the example regression equation and to regression Number Ia (Positive Design Experiment, Lot 151; Response, Pickup Weight) the ensuing discussion describes the procedure used in the interpretation of the regression results. The independent variable (X) coefficients are found under the "B Coef" column in the same order as listed in the raw data.

- 1 5.0830 (Specific Gravity)
- 2 4.7497 (Impregnation Time)
- 3 -0.2499 (Polarization Time)

To maximize plaque pickup weight during the impregnation/polarization process, the coefficients dictate a preference for the higher values of independent variables 1 and 2 (specific gravity 1.80, impregnation time 1 hour), and the lower value for independent variable Number 3 (polarization time 15 minutes). This procedure, of course, just involves mathematically maximizing the response or the Y term of the regression equation. It should also be noted the first two variables exhibits a much greater effect

upon the response of pickup weight than the third variable (polarization time).

A problem which frequently arises in an attempt to optimize the process variable levels in a study such as this is the variable levels dictated by one regression for a particular desired response may not be the same variable levels dictated by a second regression for an equally desired response. For example, the third independent variable in regression Number Ib (response, plate capacity) dictates a preference for the higher level of polarization time in contrast to the lower level preferred for pickup weight (Regression Number Ia). In this situation, engineering judgment must be applied to determine the process variable levels which will optimize the process.

To facilitate the interpretation of the various subregression of a major regression, tables were compiled summarizing the independent variable coefficients. Table Number V summarizes the coefficient of regression Number I and Table Number VI summarizes the coefficients of regression Number III. It should be remembered in the interpretation, a positive coefficient value requires an increasing independent variable value to increase the response value and a negative coefficient value requires a decreasing independent variable value to increase the response value.

TABLE V

POSITIVE STUDY COEFFICIENT SUMMATION REGRESSION I

RESPONSES	1	2	3	4	5
	PICKUP	PLATE	GR AM	PLAQUE CHARA-	PLAQUE CHARAC-
	WEIGHT	CAPACITY	CAPACITY	CTER. LOT 151	TER. AVERAGED
<u>VARIABLES</u>					,
<ol> <li>Specific Gravity of Nitrate</li> </ol>	5.0830	0.2366	0.0049	0.2499	<b>-</b> 0.0999
2. Impregnation Time	4.7497	0.2516	0.0073	0.7499	0.2499
3. Polarization Time	<b>-</b> 0.2499	0.0033	0.0008	0.2499	0.5999

Response Number 1 - Strong effect upon pickup weight, high nickel nitrate specific gravity preferred.

Response Number 2 - Strong effect upon plate capacity, high specific gravity preferred. As expected, these two responses are highly correlated. Observing the correlation matrix for regression Number I, a correlation factor of 0.975 is listed (intersection of Row 4 and Column 5.)

Response Number 3 - Positive value, high specific gravity preferred.

Response Number 4 - Positive value, high specific gravity preferred.

Response Number 5 - Negative value indicating high specific gravity is a more harsh process condition, but is the smallest coefficient in the regression indicating the variable effect is not very important in relation to the other variables.

All the coefficients are positive values dictating a preference for high impregnation times.

### VARIABLE NUMBER 3

Response Number 1 - Negative value indicating a preference for low polarization times, but exhibits the smallest effect in the pick-up weight regression.

The remaining coefficients are positive values lending support for a preference of high polarization times; but in general, the polarization time variable exhibits the least effect upon the responses considered for investigation. From a production stand point, it would appear little is to be gained from the time and expense of long polarization times.

Summarizing the preceding interpretation, the following process conditions are preferred in the positive impregnation/polarization process assuming high plaque pick-up weight in conjunction with good electrical capacity and physical appearance are the desired impregnated plaque characteristics:

- (1) High nickel nitrate specific gravity (upper experimental limit, 1.80).
- (2) High impregnation time (upper experimental limit, one hour).
- (3) Low polarization time (lower experimental limit, 15 minutes).

TABLE VI

NEGATIVE STUDY COEFFICIENT SUMMATION REGRESSION 111

RES	PONSES	1	2	3	4	5
		PICKUP	PLATE	GR AM	PLAQUE CHARA-	PLAQUE CHARAC-
		WEIGHT	CAPACITY	CAPACITY	CTER. LOT 151	TER. AVERAGED
<u>v A</u> R	IABLES					
1.	Specific Gravity of Nitrate	<b>-</b> 0.9832	-0.1841	-0.0041	-0.0000	0.1999
2.	Impregnation Time	8.9495	0.3524	<b>-</b> 0.0098	0.0000	0.0000
3.	Polarization Time	0.5499	0.0191	0.0016	<b>-</b> 0.4999	0.4999

The first four negative coefficient values dictates a preference for a low cadmium nitrate solution specific gravity. The fifth coefficient which represents all three lots of plaques is a positive value indicating that a low specific gravity is a more harsh process condition, but the plaque characterization responses do not have the same significance in the negative study as they did in the positive study. For the most part, satisfactory impregnated plaques were obtained even with the weakest plaque lot.

### VARIABLE NUMBER 2

The very strong effects on pickup weight and plate capacity dictates a preference for long impregnation times. But, there would be some sacrifice in the electrical efficiency of the plaque as indicated by the negative coefficient of response Number 3 (gram capacity).

Not considering the negative coefficient values for the plaque characterization responses (4 and 5) for the reason stated before, the first three positive coefficient values dictate a preference for the high polarization time.

Summarizing the preceding negative study interpretation, the following process conditions are preferred in the negative impregnation/polarization process assuming high plaque pick-up weight inconjunction with good electrical capacity and physical appearance are the desired impregnated plaque characteristics:

- (1) Low cadmium nitrate specific gravity (lower experimental limit, 1.80).
- (2) High impregnation time (upper experimental limit, one hour).
- (3) High polarization time (upper experimental limit, one hour).

To evaluate the effects of the different type plaques upon the responses, the coefficient summation procedure was applied to the results of regressions Number II and IV.

TABLE VII

POSITIVE STUDY COEFFICIENT SUMMATION REGRESSION II

RES	PONSES	1 PICKUP WE IGHT	2 PLATE CAPACITY	3 GRAM CAPACITY	4 PLAQUE CHARACT.
<u>VAR IABLES</u>					
1.	Specific Gravity of Nitrate	4.1996	0.1937	0.0054	0.2469
2.	Impregnation Time	6 <u>.1</u> 439	0.0631	0.0076	0.5802
3.	Polarization Time	0.2266	0.0631	0.0012	0.1791
4.	Porosity	0.1179	-0.0103	-0.0083	<b>-</b> 0.9509

Variable Number 4 defines the different type plaques; high porosity would be a characteristic of Lot 152 (low strength, high porosity), medium porosity would be a characteristic of Lot 153 (medium strength, medium porosity), and low porosity would be a characteristic of Lot 151 (high strength, low porosity). The entire summation is presented in order that the magnitude of the effect of the fourth variable may be compared to the other variables in the regressions.

### VARIABLE NUMBER 4

Response 1 - Positive value indicating high porosity desired for increased pick-up weight, but variable least important in pick-up weight regression.

Response 2 - Negative value indicating low porosity desired for increased plate capacity, again least important variable in the regression.

Response 3 - Negative value indicating low porosity (greater strength) desired for increased plate electrical efficiencies; most important variable in regression.

Response 4 - Negative value indicating low porosity desired for enhanced plaque physical characteristics (appearance) most important variable in the regression.

Summarizing, high strength, low porosity plaques (Lot 151) are to be preferred in the positive impregnation/polarization process. Some reduction in pickup capacity would have to be tolerated, but this does not appear to be significant.

TABLE VIII

### NEGATIVE STUDY COEFFICIENT SUMMATION REGRESSION IV

RES PONSES		1	2	3	4
		PICKUP	PLATE	GR AM	PLAQUE
		WEIGHT	CAPACITY	CAPACITY	CHARACT.
<u>VARIABLES</u>					,
1.	Specific Gravity of Nitrate	0.5610	-0.0727	-0.0091	0.1666
2.	Impregnation Time	7.6607	0.3083	<b>-0.</b> 0088	0.0000
3.	Polarization Time	-0.9666	<b>-0.</b> 0555	-0.0003	<b>-0.</b> 4999
4.	Porosity	1.9398	0.0810	0.0000	-0.4623

### VARIABLE NUMBER 4

Response Number 1 - Positive value indicating high porosity desired for increased pickup weight, second most important variable in the regression.

Response Number 2 - Positive value indicating high porosity desired for increased plate capacity, second most important variable in the regression.

Response Number 3 - The value here indicates porosity does not exhibit a significant effect upon the electrical efficiency or gram capacity of a plaque.

Response Number 4 - Negative value indicating low porosity desired for enhanced plaque physical characteristics (appearance), second most important variable in the regression.

Summarizing, low strength, high porosity plaques (Lot 152) are

to be preferred in the negative impregnation/polarization process.

There will be some sacrifice in the physical appearance of the plaques; but as stated before, in the case of the negative plaques, there was very little difference in the physical appearances throughout the experiments.

#### IV. DISCUSSION

1. As evidenced by the difficulty encountered during the experimental phase of the positive plaque impregnation/polarization study (poor physical integrity of impregnated plaques, flaking material, etc.), the range of the process variable levels selected for the fractional factorial design experiments did not exhibit the expected latitude. The combination of certain levels of variables such as impregnation under vacuum, high free acid content of the nitrate solution, and polarization in a caustic solution of potassium hydroxide (KOH) resulted in the destruction of the impregnated plaques.

It is suggested that in any future approach to this type of investigation, the finalization of the variable levels in the factorial design experiments be preceded by pilot experimental runs to test the more questionable levels and combinations.

2. The design experiments which were formulated and completed investigating process variables associated with the plaque impregnation/polarization process demonstrated the following process conditions are to be preferred in the optimization of the process based upon a linear multiple regression analysis of the experimental data. An assumption is made here that high pick-up weight, good electrical capacity and a smooth clean physical appearance are the desired impregnated plaque parameters.

Preferred process conditions for the positive plaque impregnation/polarization process:

- (1) High nickel nitrate specific gravity (upper experimental limit, 1.80).
- (2) High impregnation time (upper experimental limit, one hour).
- (3) Low polarization time (lower experimental limit, 15 minutes).

Preferred process conditions for the negative plaque impregnation/polarization process:

- (1) Low cadmium nitrate specific gravity (lower experimental limit, 1.80).
- (2) High impregnation time (upper experimental limit, one hour).
- (3) High polarization time (upper experimental limit, one hour).
- 3. Three types of raw plaques which varied in strength and porosity were selected for use during the experimental work in order to evaluate their effect upon the impregnation/polarization process. Additional multiple regression analysis, incorporating this fourth variable, indicated a preference for a particular type plaque to be used in a process, assuming the same desired plaque parameters as described above.

#### Positive Impregnation/Polarization Process

A high strength, low porosity (experimental limits strength 525 lbs/in<sup>3</sup> - porosity 83.3% void) plaque is preferred.

#### Negative Impregnation/Polarization Process

A low strength, high porosity (experimental limits, strength 250 lbs/in<sup>3</sup> - porosity 86.0% void) plaque is preferred.

4. Significant improvement in the physical appearance and electrical efficiency (grams capacity) of positive plaques was obtained by impregnation with a high temperature (200°F) nickel nitrate solution and without vacuum. The enclosed photograph (see Figure 4) presents two plates from the same raw plaque lot (Lot 151) impregnated under similar conditions with the exception of nitrate temperature and vacuum. Plate Number One is from Experiment 5A (nitrate temperature 200°F, vacuum 0 inches). Plate No. 2 is from Experiment 1

# POSITIVE IMPREGNATED PLATES EXPERIMENT 5A (FIRST) EXPERIMENT 1 (SECOND)

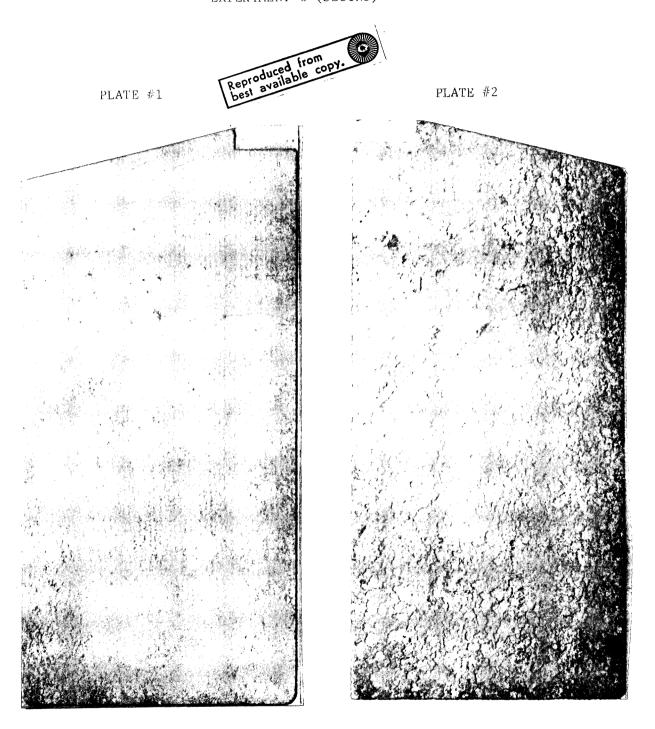


FIGURE 4

(nitrate temperature 140°F, vacuum 15 inches). Plate Number Two is observed to exhibit a very rough surface with the active material apparently caked on the outside of the plate. The suspicion that this active material would not be utilized electrically is supported by the gram capacity (active material utilization) results for the plaques in these two experiments.

# PLAQUE LOT 151 GRAM CAPACITIES

(AMPERE-HOURS/GRAMS)

]	EXPERIMENT 5A	EXPERIMENT 1
Plaque (1)	0.263	0.228
(2)	0.254	0.226
(3)	0.279	0.228

5. An interesting occurrence with respect to plaque pickup weight was observed between the two above mentioned experiments. No explanation can be given but the data will be presented for intormation. It would be expected that the plaque lot exhibiting the highest porosity (Lot 152; low strength, high porosity) would exhibit the greatest pickup weight of the three lots within an experiment. Generally this was true throughout this work with the exception of Experiment Number 1. In this case, the lowest porosity plaque Lot (151, high strength, low porosity) exhibited the greatest pickup weight, the medium porosity plaque lot (Lot 153; medium strength, medium porosity) exhibited the next highest pickup weight and the high porosity plaque lot (Lot 152) exhibited the least pickup weight.

The following is a comparison of the pickup weights (after formation) of Experiment Number 1 with Experiment Number 5A which exemplifies a more typical ratio of pickup weights.

LOT 151 PLAQUE	EXPERIMENT 1	EXPERIMENT 5A
(1)	59.9 grams	54.0
(2)	58.1	57.5
(3)	55.1	56.0
LOT 152		
(1)	43.4	66.5
(2)	43.3	65.5
(3)	41.6	56 <b>.</b> 5*
LOT 153		
(1)	50.5	54.0
(2)	51.0	59.5
(3)	53.5	59.0

<sup>\*</sup>Low value as the result of loss of material during scrubbing operation following formation process.

6. The active material utilization factor for positive plaques was increased by impregnation with a high level free acid nickel nitrate solution. Experiment 6A utilized a high free acid level nitrate solution (4 grams/liter); this may be compared with Experiment 5A which was processed under the same experimental conditions with the exception of the free acid level (1 gram/liter).

#### PLAQUE LOT 151 GRAM CAPACITIES

(AMPERE-HOURS/GRAMS)

EX	PER I	MENT 5A	EXPERIMENT 6A
Plaque	(1)	0.263	0.289
	(2)	0.254	0.297
	(3)	0.279	0.296

A question arises in connection with this practice in that the

increase in utilization may be the result of the higher free acid's attack on the sintered nickel mesh (conversion to nickel nitrate) resulting in a damaged (weaker) plaque.

It is suggested that the effect of impregnation with a high free acid level nickel nitrate solution be investigated with respect to the strength of the impregnated plaques.

- 7. Polarization in a caustic solution of potassium hydroxide (KOH) of a specific normality and with the passage of a specific current is a much more destructive environment with respect to its effect upon impregnated plaques than polarization in a caustic solution of sodium hydroxide (NaOH) with the same normality and current. No explanation can be given for this occurrence at this time, but it will be the subject of further study.
- 8. In general, it can be stated under the process variable levels selected for this study, the positive plaque impregnation/polarization process is a far more critical operation than the negative plaque impregnation/polarization process in respect to its effect upon the physical integrity of the impregnated plaques. In reference to the weaker plaque lot (Lot 152, low strength, high porosity), few positive experiments produced impregnated plaques suitable for the measurements of the various responses and none would have been suitable for use in cells. On the other hand, all the negative experiments except one produced satisfactory impregnated plaques from the weaker raw plaque lot.
- 9. The width and thickness dimensions of plate Number One were recorded for selected positive and negative plaques before and after the impregnation/polarization and formation process. As mentioned earlier, this effort was instituted to determine if there was any significant growth in the plates as a result of the process.

No discernable growth was noted with the exception of a small increase in thickness as the result of impregnation with active material.

The actual measured dimensions may be found in Appendix B of this report.

10. A straightforward review of the data did not reveal any significant trends in pick-up weight, electrical capacity, or physical appearance in respect to a plate's position on a plaque or a plaque's position in the process tank.

#### V. CONCLUSIONS AND RECOMMENDATIONS

- 1. Because of the difficulty encountered during the experimental phase of the positive plaque impregnation/polarization process study, it is recommended that in any future approach to this type of investigation, the finalization of the variable levels in the factorial design experiment be preceded by pilot experimental runs to test the more questionable levels and combinations.
- 2. As a result of the process variable investigation, the following variable levels were determined to be preferred in achieving high plaque pick-up weight inconjunction with good electrical capacity and a smooth clean physical appearance:

#### Positive Impregnation/Polarization Process

- (1) High nickel nitrate specific gravity preferred (upper experimental limit, 1.80).
- (2) High impregnation time preferred (upper experimental limit, one hour).
- (3) Low polarization time preferred (lower experimental limit, 15 minutes).

#### Negative Impregnation/Polarization Process

- (1) Low cadmium nitrate specific gravity preferred (lower experimental limit, 1.80).
- (2) High impregnation time preferred (upper experimental limit, one hour).
- (3) High polarization time preferred (upper experimental limit, one hour).
- 3. The following raw plaques characteristics (parameters, strength and porosity) were determined to be preferred in the two processes assuming the same impregnated plaque qualities described above:

#### Positive Impregnation/Polarization Process

High strength, low porosity plaques preferred (experimental limits, strength 525 lbs/in<sup>3</sup> - porosity 83.3% void).

#### Negative Impregnation/Polarization Process

Low strength, high porosity plaques preferred (experimental limits, strength 250 lbs/in<sup>3</sup> - porosity 86.0% void).

- 4. A significant improvement in the physical appearance and electrical efficiency (gram capacity) of positive plaques were obtained by impregnation with a high temperature (200°F) nickel nitrate solution without the use of vacuum.
- 5. The active material utilization factor or efficiency (gram capacity) of the positive impregnated plaques was increased by impregnation with a high level free acid nickel nitrate solution (4 grams/liter).

It is recommended that this phenomenon be further investigated by determining the degree of attack on the nickel nitrate by the higher free acid levels through strength measurements of impregnated plaques.

6. The performance of the polarization operation in a caustic solution of NaOH results in superior impregnated positive plaques (stronger, smooth and clean physical appearance) in comparison to polarization in a caustic of KOH (polarization rate 0.4 amps/in<sup>2</sup>).

It is recommended this area of the program receive additional effort to determine why KOH caustic solutions exhibit a more destructive effect upon the impregnated plaques.

- 7. In general, the positive impregnation/polarization process is a far more critical operation than the negative in respect to the range of the process variable levels which may be used.
- 8. No significant dimensional growth in the individual plates or on impregnated plaques could be discerned as a result of the impregnation/polarization process.

9. No discernable trends in pick-up weight, electrical capacity, or physical appearance with respect to a plate's position on a plaque or a plaque's position in the process tank were evident.

#### VI. PROGRAM PLAN

- 1. Additional information will be gained concerning the impregnation/polarization process during the next period by subjecting the
  resulting impregnated plaques to a scanning electro micrographic analysis.
  Through the examination of the resulting photomicrographs, it is hoped
  to determine the distribution of the active materials within the plaque
  pores and to gain a comparative knowledge of the porosity of the various
  impregnated plaques.
- 2. An experiment will be formulated and completed during the next period to determine why the polarization in a caustic solution of potassium hydroxide (KOH) presents a much more destructive environment to the impregnated plaque than polarization in a caustic solution of sodium hydroxide (NaOH). The areas of interest will be the viscosity of the two solutions and their gassing characteristics.
- 3. Work will also begin during the next period in the characterization of nickel-cadmium cell separator materials. The first step will be to investigate the organic impurity levels associated with the various materials; this will be followed by an investigation of the inorganic impurity levels. In addition to these first two steps, the materials will be subjected to the scanning electron micrographic analysis mentioned before to determine what this technique may reveal.
- 4. The following reporting period will also include work in the characterization of the nickel-cadmium cell auxiliary or "signal" electrode. It is planned to begin the investigation with the fabrication of standard signal electrodes incorporating cells modified to accept an external source of oxygen. The signal emanating from the electrode as a function of a controlled oxygen pressure will then be measured.

5. The implementation of the fraction factorial design experiment (defined in the previous Quarterly Report) formulated for the purpose of investigating the process variables associated with the electrochemical cleaning or formation process will begin in the following period. The work will begin with the processing of impregnated test plaques using impregnation/polarization processes incorporating the knowledge gained from the studies described in this report and the Third Quarterly Report.

#### REFERENCES

- (1) Second Quarterly Report, Study of Process Variables Associated With Manufacturing Hermetically-Sealed Nickel-Cadmium Cells, Eagle-Picher Industries, Inc., Joplin, Missouri, December, 1970; prepared for NASA/GSFC, Contract Number NAS5-21159.
- (2) Third Quarterly Report, Study of Process Variables Associated With Manufacturing Hermetically-Sealed Nickel-Cadmium Cells, Eagle-Picher Industries, Inc., Joplin, Missouri, June, 1971; prepared for NASA/GSFC, Contract Number NAS5-21159.
- (3) Gardner, Donald S., <u>Multiple Regression Program/1130</u>, IBM 1130-13.6 .003.

# APPENDIX A

INDIVIDUAL PLAQUE POLARIZATION VOLTAGES

The plaque polarization voltage with respect to a reference electrode was recorded for each cycle of each experiment at the beginning and end of a polarization period. In all cases, the plaque voltage with respect to the reference electrode was a negative value and the negative sign "-" will be dropped in the presentation of the data. The three plaque lots were introduced into the impregnation/polarization tank in a standard order and the following index identifies the location of each lot:

	<u>LOT 151</u>	LOT 152	LOT 153
PLAQUE	1	4	7
	2	5	8
	3	6	9

In addition, the voltage of one plaque was continuously monitored on a recorder during the polarization cycles of positive impregnation (polarization Experiment Number 4) (see Figure 5). This information was obtained to evaluate the voltage fluctuations during the polarization period. The actual recorder tracing may be found with positive Experiment Number 4 data in this Appendix.

# REFERENCE ELECTRODE - NEGATIVE PLATE

	10	BI BROWNOD BEBOINGE				
OTTOT D. 1						
CYCLE 1						
		BEGIN				
	1.	0.543	4.	0.579	7.	0.555
	2.	0.576	5.	0.585	8.	0.579
				0.544	9.	0.570
•	3,	0.580	6.	0.544	9.	0.570
	•					
		END				
	1.	0.654	4.	0.687	7.	0.625
	2.	0.677	5.		8.	0.680
	3.	0.686	6.	0.665	9.	0.670
CYCLE 2						
		BEGIN				
	1.	0.725	4.	0.760	7.	0.722
	2.	0.770	5.		8.	0.740
	3.	0.770	6.	0.715	9.	0.727
		END				
	1.	0.570	4.	0.629	7.	0.530
					8.	0.635
	2.	0.625	5.			
	3.	0.630	6.	0.590	9.	0.620
•				•		
CYCLE 3						
<del></del>		BEGIN				
		-		•		
	1.	0.650	4.	0.680	7.	0.660
	2.	0.690	5.		8.	0.686
	3.	0.689	6.	0.630	9.	0.670
		END				
	1.	0.629	4.	0.675	7 '	0.590
		0.670	5.	0.683		0.678
	2.				8.	
	3.	0.674	6.	0.639	9.	0.665
CYCLE 4						
		BEGIN				
		22021				
	7	0 620	1.	0 600	7	n 600
	1.	0.638	4.	0.690	7.	0.688
	2.	0.687	5.	0.699	8.	0.715
	3.	0.690	6.	0.650	9.	0.706
		END				
	1	0.719	/.	0.773	7	0.690
	1.	0.718	4.		7.	
	2.	0.760	5.	0.778	8.	0.760
	3.	0.770	6.	0.736	9.	0.746
	-		-			

REFERENCE ELECTRODE - NEGATIVE PLATE (CYCLE 1)
REFERENCE ELECTRODE - POSITIVE PLATE (CYCLES 2, 3 & 4)

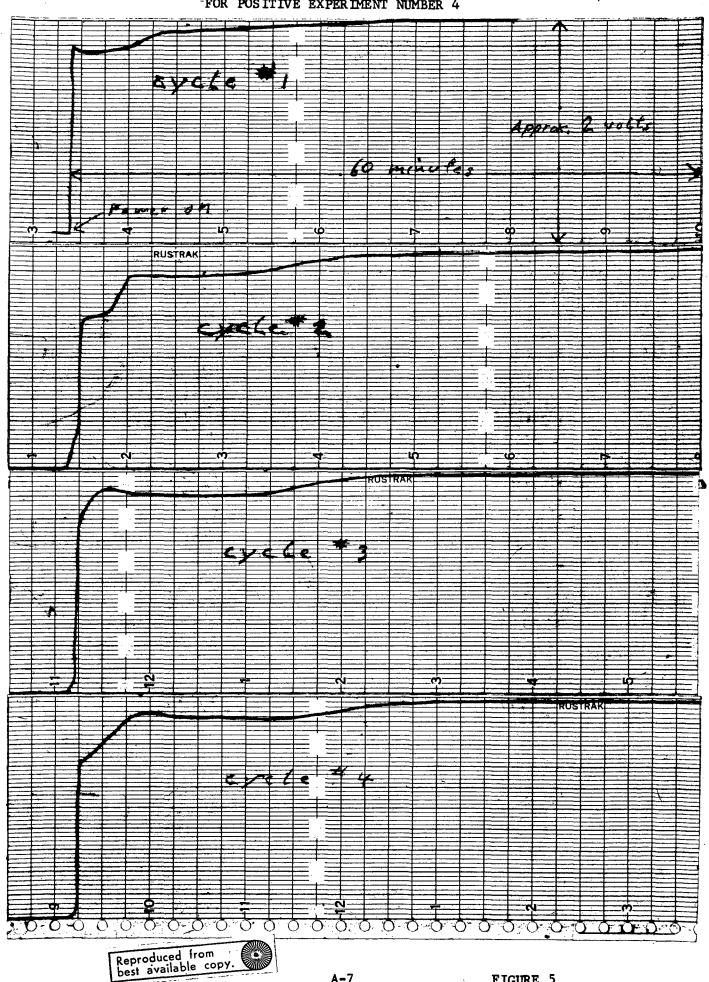
CYCLE 1						
		BEGIN				
	1.	0.393		0.445	7.	0.360
	2. 3.	0.439 0.450		0.449 0.404	8. 9.	0.434 0.429
	٠.		٠.	0.704	,.	0.427
		END				
		0.494		0.540	7.	0.470
		0.529 0.536		0.538 0.506	8. 9.	0.522 0.516
CVCTE 2						
CYCLE 2		BEGIN				
	1.	1.495	4.	1.576	7.	1.535
	2.	1.570		1.597	8.	1.596
	3.	1.586	6.	1.546	9.	1.587
		END				
	1.	1.648	4.	1.695	7.	1.625
	2.	1.690		1.707	8.	1.680
	3.	1.695	6.	1.659	9.	1.670
CYCLE 3						
		BEGIN				
	1.	1.387	4.	1.440	7.	1.390
	2.	1.420		1.440	8.	1.420
	3.	1.430	6.	1.400	9.	1.417
		END				
		1.650		1.704	7.	1.629
		1.687		1.700		1.687
	<b>3.</b>	1.696	6.	1.660	9.	1.675
CYCLE 4		DECTY				
		BEGIN				
		1.390		1.448	7.	
		1.428		1.453	8.	
;	3.	1.440	6.	1.409	9.	1.435
		END				
		1.658	4.	1.707	7.	1.636
		1.689	5.	1.710	8.	
	3.	1.700	6.	1.667	9.	1.680
			A-4			

CYCLE 1	PERO TA				
•	BEGIN				
1.	. 1.630	4.	1.600	7.	1.630
2.		5.		8.	1.630
3	. 1.640	6.	1.620	9.	1.630
	END				
1.		4.		7.	1.690
2.		5.		8.	1.690
3.	. 1.680	6.	1.670	9.	1.690
CYCLE 2					
	BEGIN				
1.		4.		7.	
2.		5.			1.470
3.	. 1.480	6.	1.470	9.	1.470
	END				
1.	. 1.630	4.	1.620	7.	1.630
2.		5.		8.	1.630
3.	. 1.630	6.	1.620	9.	1.630
CYCLE 3					
	BEGIN				
1.	. 1.470	4.	1.520	7.	1.540
2.	. 1.470	5.	1.530	8.	1.550
3.	. 1.490	6.	1.530	9.	1.550
	END				
1.	. 1.620	4.	1.590	7.	1.630
2.	. 1.620	5.			1.620
3.	. 1.630	6.	1.570	9.	1.630
CYCLE 4					
	BEGIN				
1.	1.466	4.	1.457		1.476
2		5.			1.490
3.	. 1.470	6.	1.450	9.	1.477
	END				
1.	. 1.625	4.	1.620	7.	1.590
2.		5.			1.590
3.	. 1.630	6.	1.608	9.	1.630
		A-5	i e		

# REFERENCE ELECTRODE - POSITIVE PLATE

CYCLE 1		BEGIN				
		DHOTH				
	1.	1.371	4.	1.380		1.400
	2.	1.380	<b>5.</b>		8.	1.410
	3.	1.380	6.	1.370	9.	1.400
		END				
		END				
	1.	1.710	4.	1.710	7.	1.710
	2.	1.716	5.		8.	1.710
	3.	1.710	6.	1.700	9.	1.710
CYCLE 2						
OTOLE Z		BEGIN				
	1.	1.377	4.			1.370
	2.	1.385	5.		8.	1.370
	3.	1.378	6.	1.378	9.	1.370
		END				
	1.	1.616	4.			1.610
	2.	1.620	5.			1.615
	3.	1.615	6.	1.608	9.	1.613
CYCLE 3						
		BEGIN				
	1	1 / 20	,	1 /27	7	1 / 26
	1. 2.	1.430 1.420	4. 5.		8.	1.426 1.419
	3.	1.410	6.		9.	1.426
	J.	1:410	••	11400	•	10.120
		END				
	1.	1.617	4.	1.627	7.	1.619
		1.625		1.640		1.625
	3.	1.619	6.	1.617	9.	1.625
OVOT E /						
CYCLE 4		BEGIN				
		22021				
	1.	1.474		1.438		1.436
		1.480	5.			1.450
	3.	1.480	6.	1.425	9.	1.445
		END				
	1.	1.590	4.			1.595
	2.	1.597	5.		8.	1.598
	3.	1.590	6.	1.605	9.	1,600

A-6



CYCLE 1						
		BEGIN				
	1.	1.356	4.			1.380
	2.	1.360	5.	1.360	8.	1.369
	3.	1.360	6.	1.370	9.	1.380
		END				
	1.	1.747	4.			1.740
	2.	1.750	5.	1.755		1.753
	3.	1.745	6.	1.758	9.	1.760
CYCLE 2						
		BEGIN				
	1.	1.440	4.	1.455	7.	1.437
	2.	1.440	5.			
	3.	1.446	6.	1.459	9.	1.460
		END				
	1.	1.690	4.	1.690	7.	1.684
	2.	1.698	5.	1.696	8.	1.697
	3.	1.699	6.	1.704	9.	1.708
CYCLE 3						
		BEGIN				
	1.	1.405	4.	1.419	7.	1.400
	2.	1.420	5.		8.	1.410
	3.	1.420	6.	1.420	9.	1.420
		END				
	1.	1.674	4.	1.689	7.	1.669
	2.	1.680	5.	1.680	8.	1.68 <b>0</b>
	3.	1.680	6.	1.687	9.	1.687
CYCLE 4		PROTE				
		BEGIN				
	1.	1.420	4.	1.427	7.	1.419
	2.	1.428	5.	1.420	8.	
	3.	1.430	6.	1.428	9.	1.440
		END				
	1.	1.669	4.	1.679	7.	1.660
	2.	1.676	5.	1.670	8.	1.670
	3.	1.670	6.	1.679	9.	1.680

CYCLE 1					
	BEGIN .				
1.	1.357	4.		7.	1.357
2.	1.356	5.		8.	1.358
3.	1.350	6.	1.366	9.	1.360
	END				
1.	1.647	4.	1.644	7.	1.647
2.	1.650		1.644	8.	1.647
3.	1.657	6.	1.657	9.	1.646
CYCLE 2					
	BEGIN				
1.	1.395	4.		7.	1.398
2.	1.386	5.		8.	1.400
3.	1.325	6.	1.385	9.	1.400
	END				
1.	1.586	4.	1.600	7.	1.609
2.	1.616		1.560	8.	1.617
3.	1.603	6.	1.606	9.	1.620
CYCLE 3					
-	BEGIN				
1.	1.298	4.	1.320	7.	1.319
2.	1.325	5.	1.250	8.	1.324
3.	1.317	6.	1.327	9.	1.322
	END				
1.	1.599	4.	1.614	7.	1.607
2.	1.624	5.	1.540	8.	1.617
3.	1.617	6.	1.615	9.	1.616
CYCLE 4					
<u>010111 4</u>	BEGIN				
1.	1.394	4.	1.420	7.	1.428
2.	1.423	5.		8.	1.436
3.	1.413	6.	1.426	9.	1.433
	END				
1.	1.596	4.	1.620	7.	1.613
2.	1.620	5.	1.549	8.	1.620
3.	1.618	6.	1.620	9.	1.620
		A-9		•	

		•				
CYCLE 1		BEGIN				
	1.	1.255	4.	1.267	7.	1.260
	2. 3.	1.265 1.270	5. 6.	1.260 1.276	8. 9.	1.278 1.270
		END				
	1.	1.410	4.	1.418	7.	1.410
		1.420		1.410		1.419
	3.	1.426	6.	1.416	9.	1.419
CYCLE 2		BEGIN				
		DEGIN				
	1.	1.320	4.	1.310	7.	1.309
	2.	1.320		1.292		1.310
	3.	1.320	6.	1.310	9.	1.310
		END				
		1.400 ·		1.385		1.417
		1.415		1.350		1.420
	3.	1.420	6.	1.400	9.	1.420
CYCLE 3						
		BEGIN				
	1.	1.306	4.	1.290	7.	1.286
	2.	1.310	5.	1.266		1.296
	3.	1.316	6.	1.295	9.	1.297
		END				
	1.	1.404	4.	1.360	7.	1.386
		1.417	5.		8.	1.387
	3.	1.418	6.	1.364	9.	1.378

				•		
CYCLE 1						
	BE	GIN				
		200			-	1 200
		330	4. 1.370		7.	1.380
		350	5. 1.370 6. 1.380		8. 9.	1.370 1.380
	3. 1.	370	6. 1.380		7.	1.300
	E	ND				
	1. 1.	570	4. 1.570		7.	1.570
	2. 1.	570	5. 1.570		8.	1.570
	3. 1.	570	6. 1.570		9.	1.570
CYCLE 2						
CICHE 2	BE	GIN				
			,		_	1 000
		300	4. 1.310		7.	1.300
		300	5. 1.300		8.	1.300
	3. 1.	300	6. 1.310		9.	1.300
	E	ND				
	1. 1.	580	4. 1.590		7.	1.580
		590	5. 1.590		8.	1.580
		580	6. 1.590		9.	1.590
OVOI D 2						
CYCLE 3	BE	GIN				
	50					
	1. 1.	330	4. 11.340		7.	1.340
	2. 1.	340	5. 1.340		8.	1.340
	3. 1.	340	6. 1.350		9.	1.340
	E	ND				
	1 7	600	/ 1 600		7	1 610
	1. 1. 2. 1.	600	4. 1.600 5. 1.610			1.610 1.600
		610	6. 1.610		9.	1.610
	J. 1.	010	0. 1.010			1.010
CYCLE 4						
	BE	GIN				
	1. 1.	300	4. 1.310		7.	1.320
		310	5. 1.310		8.	1.320
		310	6. 1.320		9.	1.320
	E	ND				
		540	4. 1.550		7.	1.550
		550	5. 1.550		8.	1.550
	3. 1.	550	6. 1.550		9.	1.550
			A-11			

CYCLE 1						
		BEGIN				
1	1.	1.300	4.	1.310	7.	1.300
	2.	1.300	5.	1.300	8.	1.300
	3.	1.300	6.	1.300	9.	1.300
	-					
		END				
1	1.	1.440	4.	1.440	7.	1.440
	2.	1.450	5.	1.440	8.	1.440
	3.	1.450	6.	1.440	9.	1.440
CYCLE 2		•				
<u> </u>		BEG IN				
1	1.	1.250	4.	1.270	7.	1.260
	2.	1.300	5.	1.270	8.	1.260
	3.	1.270	6.	1.270	9.	1.260
•	•	1.2.0	- •			
		END				
1	1.	1.340	4.	1.350	7.	1.350
	2.	1.370	5.	1.350	8.	1.350
	3.	1.350	6.	1.350	9.	1.350
•	•	1.330	•	1.330	•	1.550
CYCLE 3						
GIGHE 3		BEGIN				
		DEG TH				
•	1.	1.300	4.	1.300	7.	1.320
	2.	1.300	5.	1.300	8.	1.320
	2. 3.	1.300	6.	1.300	9.	1.320
•	٠.	1.300	0.	1.500	٠.	1.320
		EXT				
		END				
•	1	1 270	4.	1.380	7	1.380
	1.	1.370	_			1.380
	2.	1.400	5. 6.	1.380	8. 9.	1.380
-	3.	1.380	0.	1.380	7.	1.300
ONOT D. /						
CYCLE 4		DECTN				
		BEGIN				
4	1	1.270	4.	1.270	7.	1.310
	1.		5.	1.260	8.	1.310
	2.	1.300				
	3.	1.300	6.	1.280	9.	1.320
		END				
		UMD				
1	1.	1.400	4.	1.400	7.	1.400
	2.	1.420	5.	1.390	8.	1.400
	3.	1.420	6.	1.400	9.	1.400
•	•	I. TLU	•	1.700	, .	1.400

# EXPERIMENT NUMBER 10B

CYCLE 1		BEGIN				
	1. 2. 3.	1.290 1.290 1.290		1.290 1.290 1.300	7. 8. 9.	1.310 1.300 1.300
		END				
	1. 2. 3.	1.640 1.650 1.640	5.	1.640 1.640 1.640		1.640 1.650 1.650
CYCLE 2		BEGIN				
	1. 2. 3.	1.300 1.300 1.310	5.	1.300 1.300 1.310		1.310 1.310 1.310
		END				
	1. 2. 3.	1.540 1.550 1.550	5.	1.540 1.540 1.550	7. 8. 9.	1.570 1.560 1.570
CYCLE 3		BEGIN				
	1. 2. 3.	1.300 1.310 1.310		1.320 1.320 1.320		1.340 1.340 1.350
		END				
		1.520 1.520 1.530		1.520 1.520 1.530		1.540 1.540 1.540
CYCLE 4		BEGIN				
	1. 2. 3.	1.420 1.430 1.430	4. 5. 6.	1.430 1.430 1.440	7. 8. 9.	1.440 1.450 1.460
		END			_	
	1. 2. 3.	1.550 1.560 1.560	4. 5. 6.	1.560 1.550 1.560	7. 8. 9.	1.570 1.570 1.570

CYCLE 1	PRO TV				
	BEGIN				
1.	1.370	4.	1.370	7.	1.380
2.	1.380	5.	1.370	8.	1.390
3.	1.380	6.	1.370	9.	1.390
	2,000	- •	2.5		
	END				
1.	1.570	4.	1.570	7.	1.560
2.	1.580	5.	1.570	8.	1.570
3.	1.580	6.	1.570	9.	1.570
CYCLE 2					
	BEGIN				
1.	1.370	4.	1.400	7.	1.430
2.	1.390	5.	1.410	8.	1.440
3.	1.400	6.	1.420	9.	1.440
5.	10.100	•	20.120		20.10
	END				
1.	1.570	4.	1.570	7.	1.570
2.	1.580	5.	1.580	8.	1.580
3.	1.570	6.	1.580	9.	1.580
J.	1.570	٠.	1.500	٠,	1.500
CYCLE 3					
	BEGIN				
1.	1.370	4.	1.380	7.	1.370
2.	1.380	5.	1.380	8.	1.370
3.	1.370	6.	1.380	9.	1.380
	END				
1.	1.580	4.	1.580	7.	1.580
2.	1.590	5.	1.590	8.	1.590
3.	1.580	6.	1.590	9.	1.590

CYCLE 1						
		BEGIN				
	1.	1.350	4.	1.350	7.	1.360
	2.	1.360	5.	1.360	8.	1.360
	3.	1.350	6.	1.360	9.	1.370
		END				
	1.	1.380	4.	1.390	7.	1.380
	2.	1.380	5.	1.400	8.	1.390
	3.	1.390	6.	1.400	9.	1.400
CYCLE 2						
		BEGIN				
	1.	1.350	4.	1.360	7.	1.370
	2.	1.360	5.	1.360	8.	1.370
	3.	1.360	6.	1.370	9.	1.370
		END				
	1.	1.390	4.	1.400	7.	1.390
	2.	1.400	5.	1.410	8.	1.400
	3.	1.400	6.	1.410	9.	1.400
CYCLE 3						
		BEGIN				
	1.	1.350	4.	1.400	7.	1.400
	2.	1.390	5.	1.410	8.	1.410
	3.	1.400	6.	1.420	9.	1.420
		END				
	1.	1.470	4.	1.470	7.	1.460
	2.	1.470	5.	1.480	8.	1.470
	3.	1.470	6.	1.480	9.	1.480

CYCLE 1		BEGIN				
		DEGIN				
	1.	1.320	4.	1.320	7.	1.330
	2.	1.320	5.	1.320	8.	1.330
	3.	1.320	6.	1.330	9.	1.330
		END				
	1.	1.500	4.	1.490	7.	1.490
	2.	1.500	5.	1.490	8.	1.490
	3.	1.500	6.	1.500	9.	1.500
			•			
CYCLE 2		•				
		BEGIN				
	1.	1.300	4.	1.320	7.	1.340
	2.	1.300	5.	1.330	8.	1.340
	3.	1.310	6.	1.330	9.	1.330
		END				·
	1.	1.510	4.	1.490	7.	1.510
	2.	1.520	5.	1.500	8.	1.500
	3.	1.520	6.	1.500	9.	1.500
				2,500	•	1,300
CYCLE 3						
		BEGIN				
	1.	1.390	4.	1.400	7.	1.400
	2.	1.400	5.	1.410	8.	1.400
•	3.	1.400	6.	1.420	9.	1.400
	<i>J</i> .	1.400	•	1.720	٠,	
		END				
	1.	1.570	4.	1.550	7.	1.560
	2.	1.580	5.	1.550	8.	1.550
	3.	1.580				
	J.	1.300	6.	1.550	9.	1.570

CYCLE 2					
	BEGIN				
1 2 3	. 1.300	4. 5. 6.	1.316 1.320 1.330	7. 8. 9.	1.326 1.346 1.339
	END				
1 2 3	1.335	4. 5. 6.	1.386 1.390 1.390	7. 8. 9.	1.376 1.380 1.384
CYCLE 3					
	BEGIN				
1 2 3	1.332	4. 5. 6.	1.360 1.369 1.370	7. 8. 9.	1.360 1.370 1.369
	END				
1 2 3	1.402	4. 5. 6.	1.407 1.410 1.410	7. 8. 9.	1.397 1.400 1.405

# APPENDIX B

PLAQUE DIMENSIONAL MEASUREMENTS

The following dimensions were taken from plate Number One (See Figure 1) of a plaque in all cases. The recorded values are the results of a single measurement in the mid portion of the subject plate.

# POSITIVE EXPERIMENT 1

7.0m 151		WIDTH	THICKNESS
LOT 151 (Start)	1	2.678"	0.030"
(Scarc)	2	2.679"	0.030"
	3	2.672"	0.030"
	_		
(After	1	2.683"	0.035"
Polarization)	2	2.683"	0.035"
•	3	2.677"	0.034"
(After	1	2.675"	0.032"
Formation)	2	2.679"	0.031"
102	3	2.675"	0.031"
150			
LOT 152	1	2.675"	0.033"
(Start)	1 2	2.673"	0.035
	3	2.671"	0.030"
	3	2.0/1	0.030
(After	1	2.694"	0.034"
Polarization)	2	2.685"	0.031"
	3	2.686"	0.030"
(After	1	2.669"	0.032"
Formation)	2	2.678"	0.028"
rormacion,	3	2.675"	0.028"
150			
LOT 153 (Start)	1	2.677"	0.031"
(Start)	2	2.670"	0.027"
	3	2.672"	0.030"
	_		
(After	1	2.688"	0.035"
Polarization)	2	2.683"	0.035"
	3	2.679"	0.035"
(After	1	2.675"	0.033"
Formation)	2	2.670"	0.030"
I OTHECTORY	3	2.675"	0.030"
	_	<del></del>	

#### POSITIVE EXPERIMENT 2

LOT 151		<u>WIDTH</u>	THICKNESS
(Start)	1	2.675"	0.030"
	2	2.670"	0.030"
	3	2.671"	0.030"
(After	1	2.675"	0.031"
Polarization)	2	2.680"	0.031"
	3	2.680"	0.031"
(After	1	2.671"	0.030"
Formation)	2	2.675"	0.030"
	3 .	2.675"	0.030"
LOT 152			
(Start)	1	2.675"	0.030"
	2	2.671"	0.030"
	3	2.669"	0.030"
(After	1	2.687"	0.032"
Polarization)	2	2.690"	0.032"
	3	2.681"	0.031"
(After	1	2.676"	0.031"
Formation)	2	2.676"	0.030"
•	3	2.673"	0.030"
LOT 153			
(Start)	1	2.680"	0.030"
•	2	2.670"	0.030"
	3	2.675"	0.031"
(After	1	2.686"	0.031"
Polarization)	2	2.685"	0.031"
·	3	2.680"	0.031"
(After	1	2.677"	0.030"
Formation)	2	2.675"	0.030"
	3	2.680"	0.030"

#### POSITIVE EXPERIMENT 3

LOT 151		WIDTH	THICKNESS
(Start)	1 2 3	2.680" 2.675" 2.675"	0.030" 0.030" 0.029"
(After Polarization)	1 2 3	2.675" 2.677" 2.683"	0.035" 0.033" 0.032"
(After Formation)	1 2 3	2.675" 2.677" 2.679"	0.032" 0.032" 0.032"
LOT 152 (Start)	1 2 3	2.670" 2.675" 2.675"	0.030" 0.030" 0.030"
(After Polarization)	1 2 3	2.679" 2.675" 2.675"	0.025"* 0.025" 0.025"
(After Formation)	1 2 3	*	*
LOT 153 (Start)	1 2 3	2.675" 2.675" 2.675"	0.030" 0.030" 0.030"
(After Polarization)	1 2 3	2.675" 2.675" 2.675"	0.035" 0.035" 0.032"
(After Formation)	1 2 3	k	*

<sup>\*</sup>The flaking and subsequent loss of the nickel matrix and active material resulted in the thickness reduction of Lot 152 and prevented the measurement of dimensions after formation for Lots 152 and 153.

# NEGATIVE EXPERIMENT 1

LOT 151		LENGTH	THICKNESS
(Start)	1	5.515"	0.028 - 0.031"
(Start)	2	5.515"	0.029 - 0.030"
	3	5.515"	0.028 = 0.029"
	3	3.313	0.020 - 0.027
(After	1	5.517"	0.031"
Polarization)	2	5.520"	0.030"
·	3	5.520"	0.030"
/ A Strong	1	5.520"	0.030"
(After	2	5.520"	0.030"
Formation)	3	5.519"	0.030"
	3	2.213	0.030
LOT 152		-	
(Start)	1	5.520"	0.029" - 0.031"
•	2	5.525"	0.029 - 0.030"
	3	5.525"	0.028 - 0.029"
( A <b>f</b> & a w	1	5.520"	0.033"
(After		5.518"	0.034"
Polarization)	2	5.521"	0.034
	3	5.321"	0.033
(After	1	5.517"	0.032
Formation)	2	5.515"	0.031"
-	3	5.516"	0.030"
LOT 153			
(Start)	1	5.518"	0.027 - 0.030"
(bcarc)	2	5.518"	0.027 - 0.030"
	3	5.525"	0.025 - 0.030"
	3	J. 323	04023
(After	1	5.517"	0.030
Polarization)	2	5.525"	0.032"
	3	5.520"	0.030"
( A E+o~	1	5.519"	0.031"
(After		5.520"	0.030"
Formation)	2 3		0.030"
	3	5.520"	0.030

### NEGATIVE EXPERIMENT 2

LOT 151 (Start) 1 5.515" 0.023 2 5.515" 0.023 3 5.515" 0.023	8" 8" 0"
	8" 0"
3 5.515" 0.026	ייכ
(After 1 5.519" 0.036	
Polarization) 2 5.518" 0.030	
3 5.519" 0.029	∌"
(After 1 5.520" 0.03	
Formation) 2 5.516" 0.030	
3 5.519" 0.036	)"
LOT 152	
(Start) 1 5.515" 0.029	<b>)''</b> .
2 5.515" 0.029	∌"
3 5.518" 0.036	)"
(After 1 5.520" *	
Polarization) 2 5.518"	
3 5.520"	
(After 1 5.518" *	
Formation) 2 5.518"	
3 5.518"	
LOT 153	
(Start) 1 5.518" 0.02	9"
2 5.518" 0.02	911
3 5.518" 0.030	)"
(After 1 5.520" 0.03	יי כ
Polarization) 2 5.519" 0.03	)"C
3 5.520" 0.03	)"
(After 1 5.520" 0.03	ייכ
Formation) 2 5.519" 0.03	
3 5.516" 0.03	

<sup>\*</sup>The impregnated plaques of Lot 152 were so badly warped that an accurate thickness measurement could not be taken.

## APPENDIX C

POSITIVE IMPREGNATION/POLARIZATION EXPERIMENT

VARIABLE LEVELS

	VARIABLE	LEVEL
1.	Specific Gravity of Nitrate	1.70
2.	Free Acid (Controlled By Addition of HNO3)	1.0 gms/liter
3.	Temperature of Nitrate (In I/P Tank)	132 to 137°F
4.	Time of Impregnation	1 Hour
5.	Vacuum	15 Inches
6.	Wash Time	30 Minutes
7.	Wash (Number of Cycles)	1 Cycle
8.	Wash Water Temperature	59°F
9.	pH of Wash Water (Measured pH Paper)	4.5 to 5.3
10.	Type of Caustic	NaOH
11.	Specific Gravity of Caustic	1.30
12.	Temperature of Caustic (In I/P Tank)	64 to 74°F
13.	Amount of NH <sub>3</sub> in Caustic	0.021 to 0.037 N
14.	Amount of CO <sub>3</sub> in Caustic	0.2 N
15.	Amount of OH in Caustic	9.2 N
16.	Polarization Current	$0.4 \text{ amps/in}^2$
17.	Polarization Time	1 Hour
18.	Voltage of Plaque to Reference	See Appendix
19.	Amount of Cycles with Same Caustic	1 Cycle
20.	Number of Total Cycles	4 Cycles
21.	Type of Plaque	3 Types

	<u>VARIABLE</u>	<u>LEVEL</u>
1.	Specific Gravity of Nitrate	1.70
2.	Free Acid (Controlled By Addition of HNO3)	1.2 to 1.4 gms/liter
3.	Temperature of Nitrate (In I/P Tank)	136 to 145°F
4.	Time of Impregnation	15 Minutes
5.	Vacuum	0 Inches
6.	Wash Time	10 Minutes
7.	Wash (Number of Cycles)	1 Cycle
8.	Wash Water Temperature	64 to 70°F
9.	pH of Wash Water (Measured pH Paper)	4.5 to 5.0
10.	Type of Caustic	кон
11.	Specific Gravity of Caustic	1.20
12.	Temperature of Caustic (In I/P Tank)	140 to 151°F
13.	Amount of NH <sub>3</sub> in Caustic	0.008 to 0.013 N
14.	Amount of CO <sub>3</sub> in Caustic	0.24 to 0.44 N
15.	Amount of OH in Caustic	0.50 to 0.55 N
16.	Polarization Current	$0.4 \text{ amps/in}^2$
17.	Polarization Time	1 Hour
18.	Voltage of Plaque to Reference	See Appendix
19.	Amount of Cycles with Same Caustic	4 Cycles
20.	Number of Total Cycles	4 Cycles
21.	Type of Plaque	3 Types

		<u>v A</u> r I	[ABLI	<u>E</u>		LEVEL
1.	As	shown	for	previous	experiments	1.70
2.	11	11	11	**	11	1.0 gms/liter
3.	11	**	11	77	II	175 to 189°F
4.	**	**	11	11	11	15 Minutes
5.	11	11	11	11	II	15 Inches
6.	"	**	. 11	**	11	10 Minutes
7.	"	11	11	fŧ	11	3 Cycles
8.	11	11	11	11	11	52 to 54°F
9.	11	11	11	**	11	4.5 to 5.3
10.	11	11	11	11	11	кон
11.	11	***	11	tt	11	1.30
12.	"	"	11	11	11	150 to 158°F
13.	"	11	11	***	11	0.010 to 0.015 N
14.	11	11	11	***	11	0.84 to 1.40 N*
15.	11	***	"	**	11	7.1 to 7.4 N
16.	11	***	11	ff	11	$0.4 \text{ amps/in}^2$
17.	11	11	11		II	15 Minutes
18.	11	11	11	Ħ	11	See Appendix
19.	11	11	11	11	II	4 Cycles
20.	11	11	11	11	11	4 Cycles
21.	11	**	11	11	II.	3 Types

<sup>\*</sup>The high carbonate contamination indicated here is suspected to be the result of the use of carbonate contaminated deionized water.

		VAR]	ABLI	<u>3</u>		LEVEL
1.	As	shown	for	previous	experiments	1.70
2.	11	11	"	"	tt	1.0 gms/liter
3.	11	***	11	11	11	180 to 188°F
4.	11	**	11	"	H	1 Hour
5.	11	**	11	ff	II	15 Inches
6.	11	**	11	11	tt	10 Minutes
7.	11	**	11	11	Ħ	3 Cycles
8.	**	11	11	**	ff.	50 to 56°F
9.	11	***	11	**	Ħ	4.5
10.	"	11	11	**	Ħ	КОН
11.	11	***	11	11	II	1.30
12.	ff	11	"	11	11	150 to 156°F
13.	"	11	***	**	Ħ	0.010 to 0.016 N
14.	11	11	11	**	11	0.44 to 0.60 N
15.	11	11	11	11	ff	7.0 to 7.4 N
16.	"	11	11	FF.	tt	$0.4 \text{ amps/in}^2$
17.	11	11	**	**	***	1 Hour
18.	11	11	"	71	11	See Appendix
19.	11	11	"	11	Ħ	4 Cycles
20.	"	11	**	11	11	4 Cycles
21.	11	11	**	. 11	II	3 Types

		VAR I	BLE	<u>5</u>		LEVEL
1.	As	shown	for	previous	experiments	1.70
2.	11	**	11	11	tt	0.80 to 1.0 gm/liter
3.	"	11	11	11	Ħ	178 to 186°F
4.	11	11	11	11	II	1 Hour
5.	11	**	11	11	Tf	0 Inches
6.	"	11	11	11	11	10 Minutes
7.	11	**	11	11	11	3 Cycles
8.	"	*1	11	11	H	50 to 52°F
9.	"	11	11	"	Ħ	4.5
10.	11	11	11	11	11	NaOH
11.	11	11	11	11	11	1.30
12.	**	11	11	11	tt	150 to 160°F
13.	**	11	11	11	11	0.007 to 0.018 N
14.	"	11	11	11	11	0.28 to 0.44 N
15.	11	**	11	11	11	9.4 to 9.8 N
16.	11	*1	11	11	Ħ	$0.4 \text{ amp/in}^2$
17.	11	***	11	11	11	1 Hour
18.	11	11	11	11	11	See Appendix
19.	"	H	11	11	11	4 Cycles
20.	11	"	11	11	11	4 Cycles
21.	"	11	11	11	II	3 Types

	V	AR I ABLE	<u> </u>			LEVEL
1.	As	shown	for	previous	experiments	1.80
2.	11	11	11	11	11	3.8 gms/liter
3.	**	11	"	11	11	182 to 190°F
4.	11	11	**	11	11	1 Hour
5.	11	**	**	н	11	0 Inch
6.	11	**	11	11	11	10 Minutes
7.	**	11	11	. 11	i n	3 Cycles
8.	11	**	11	Ħ	11	48 to 50°F
9.	11	**	11	11	"	4.5
10.	11	"	11	11	11	NaOH
11.	**	**	**	!!	п	1.30
12.	11	11	11	11	11	150 to 160°F
13.	11	**	"	11	11	0.009 to 0.024 N
14.	11	**	11	11	11	0.16 to 0.24 N
15.	11	***	11	11	11	9.6 to 9.9 N
16.	11	***	11	***	f f	$0.4 \text{ amps/in}^2$
17.	11	11	**	11	11	1 Hour
18.	11	11	11	"	11	See Appendix
19.	11	11	11	n	11	4 Cycles
20.	11	11	**	11	11	4 Cycles
21.	11	11	"	"	11	3 Types

	Ž	AR IABI	E			LEVEL
1.	As	shown	for	previous	experiments	1.80
2.	"	11	"	11	11	3.4 to 4.4 gms/liter
3.	**	**	11	11	II	188 to 194°F
4.	11	**	"	11	п	15 Minutes
5.	11	**	"	**	11	0 Inches
6.	11	***	11	11	11	10 Minutes
7.	11	**	11	***	11	3 Cycles
8.	11	11	11	11	11	46 to 48°F
9.	11	11	11	11	11	4.5
10.	"	11	11	11	11	кон
11.	11	11	11	11	11	1.30
12.	11	**	11	**	11	150 to 160°F
13.	**	***	11	***	11	0.006 N
14.	**	11	11	**	11	0.12 N
15.	11	"	11	**	11	7.6 N
16.	"	11	11	11	11	$0.4 \text{ amps/in}^2$
17.	"	**	11	11	11	15 Minutes
18.	11	**	***	***	11	See Appendix
19.	11	71	11	11	"	4 Cycles
20.	11	11	11	***	11	4 Cycles
21.	11	**	11	11	11	3 Types

		<u>VAR IAI</u>	BLE			LEVEL
1.	As	shown	for	previous	experiments	1.80
2.	11	**	"	11	11	0.8 to 1.0 gms/liter
3.	11	11	11	***	11	188 to 196°F
4.	**	11	"	11	***	15 Minutes
5.	11	11	11	11	11	0 Inches
6.	11	11	"	*1	11	10 Minutes
7.	11	***	11	11	11	3 Cycles
8.	**	**	11	11	11	48 to 49°F
9.	11	11	11	11	11	4.5
10.	11	11	11	11	11	NaOH
11.	**	11	11	ff	11	1.30
12.	11	11	11	11	Tr.	150 to 170°F
13.	11	11	"	#1	11	0.014 to 0.026 N
14.	11	11	11	11	TI .	0.20 to 0.44 N
15.	11	**	**	11	11	9.2 to 9.7 N
16.	11	**	11	11	"	$0.4 \text{ amps/in}^2$
17.	11	"	11	***	11	1 Hour
18.	11	"	11	11	**	See Appendix
19.	11	11	11	11	***	4 Cycles
20.	11	"	11	***	"	4 Cycles
21.	11	"	11	11	***	3 Typės

	<u>v</u>	'AR IABI	E			LEVEL
1.	As	shown	for	previous	experiments	1.80
2.	"	11	"	**	11	1.0 to 1.2 gms/liter
3.	11	**	11	<b>f1</b>	11	186 to 190°F
4.	11	11	"	11	"	1 Hour
5.	11	11	11	11	"	0 Inch
6.	71	11	11	11	II .	10 Minutes
7.	11	11	11	11	11	3 Cycles
8.	11	"	11	11	IT	49°F
9.	*1	**	"	Ħ	11	4.5
10.	11	11	11	11	11	NaOH
11.	11	11	"	н .	***	1.30
12.	11	***	"	11	11	138 to 156°F
13.	11	11	11	11	"	0.012 to 0.014 N
14.	n	11	***	11	11	0.32 to 0.36 N
15.	*1	11	11	11	11	9.2 to 9.5 N
16.	11	11	11	11	11	$0.4 \text{ amp/in}^2$
17.	**	11	11	11	"	15 Minutes
18.	***	11	11	11	11	See Appendix
19.	"	11	"	11	"	4 Cycles
20.	**	11	11	Ħ	11	4 Cycles
21.	11	11	11	II	**	3 Types

### EXPERIMENT NUMBER 10B

		VARIA	BLE			LEVEL
1.	As	shown	for	previous	experiments	1.70
2.	11	11	11	**	11	1.0 to 1.8 gms/liter
3.	11	11	**	11	11	182 to 195°F
4.	11	11	11	"	п	15 Minutes
5.	11	tt	11	**	11	0 Inch
6.	11	**	**	11	11	10 Minutes
7.	11	11	11	11	11	3 Cycles
8.	11	11	11	11	11	48°F
9.	11	п	11	11	11	4.0
10.	11	11	"	***	11	NaOH
11.	11	11	"	II .	11	1.30
12.	11	**	11	11	IT	135 to 148°F
13.	11	11	"	"	"	0.009 to 0.010 N
14.	11	11	"	***	11	0.32N
15.	11	**	11	11	11	8.9 N
16.	11	11	"	***	11	$0.4 \text{ amps/in}^2$
17.	11	11	11	11		15 Minutes
18.	11	**	11	11	11	See Appendix
19.	11	11	11	Ħ	11	4 Cycles
20.	11	11	11	***	11	4 Cycles
21.	"	11	11	**	11	3 Types

## APPENDIX D

NEGATIVE IMPREGNATION/POLARIZATION EXPERIMENT

VARIABLE LEVELS

		VARIA	ABLES	<u>3</u>		<u>LEVELS</u>
1.	As	shown	for	previous	experiments	1.90
2.	11	11	11	**	11	0.4 to 0.5 gms/liter
3.	11	11	11	***	11 -	130 to 138°F
4.	"	**	"	11	t t	15 Minutes
5.	"	**	11	11	11	0 Inches
6.	***	11	11	**	11	10 Minutes
7.	11	11	11	11	11	3 Cycles
8.	11	11	11	**	11	48 to 52°F
9.	11	11	11	11	11	4.5
10.	11	11	11	"	11	NaOH
11.	11	11	11	11	11	1.30
12.	11	11	"	11,	11	78 to 88°F
13.	11	11	11	11	11	0.012 to 0.017 N
14.	11	11	11	11	11	0.20 to 0.28 N
15.	11	**	11	11	11	8.6 to 9.1 N
16.	11	19	11	**	II	$0.4 \text{ amps/in}^2$
17.	11	11	11	11	11	1 Hour
18.	"	11	11	11	11	See Appendix
19.	11	11	11	**	11	3 Cycles
20.	11	II	11	11	TT .	3 Cycles
21.	11	11	11	II.	"	3 Types

		VARIA	BLES			<u>LEVEL</u>
1.	As	shown	for	previous	experiments	1.90
2.	"	**	**	11	ff	0.5 to 0.6 gms/liter
3.	11	11	"	ti	11	132 to 140°F
4.	11	11	11	11	11	1 Hour
5.	11	11	11	"	11	0 Inches
6.	11	***	***	tt	11	10 Minutes
7.	11	**	11	"	II.	3 Cycles
8.	"	11	11	11	11	50°F
9.	11	11	11	11	11	4.5
10.	11	***	11	11	11	NaOH
11.	11	"	11	***	Ħ	1.30
12.	11	* **	11	***	11	78 to 94°F
13.	**	***	11	***	11	0.007 N
14.	**	11	11	**	Ħ	0.16 N
15.	11	**	11	***	11	8.3 N
16.	11	11	11	***	11	$0.4 \text{ amps/in}^2$
17.	11	11	11	***	11	15 Minutes
18.	11	"	"	11	11	See Appendix
19.	"	11	11	11	11	3 Cycles
20.	11	11	11	11	11	3 Cycles
21.	**	11	11	11	<b>11</b>	3 Types

	<u>v</u>	AR IABI	LES			<u>LEVELS</u>
1.	As	shown	for	previous	experiments	1.80
2.	11	"	**	**	11	0.4 to 0.6 gms/liter
3.	11	11	"	11	11	130 to 138°F
4.	11	11	11	***	11	1 Hour
5.	"	"	"	11	11	0 Inches
6.	11	11	11	Ħ	"	10 Minutes
7.	"	***	11	11	"	3 Cycles
8.	11	11	11	11	"	48°F
9.	"	11	n	11	11	4.5
10.	"	11	11	tt.	11	NaOH
11.	11	11	11	ff	11	1.30
12.	11	**	11	11	11	92 to 103°F
13.	11	"	11	ŧŧ	. 44	0.009 N
14.	17	11	11	11	11	0.20 to 0.92 N*
15.	11	"	11	11	11	9.0 to 9.3 N
16.	11	**	11	11	ff	$0.4 \text{ amps/in}^2$
17.	"	**	11	11	11	1 Hour
18.	11	11	11	11	**	See Appendix
19.	11	11	11	11	11	3 Cycles
20.	"	**	11	***	11	3 Cycles
21.	11	**	11	H	Ħ	3 Types

<sup>\*</sup>Suspect high carbonate indication is the result of the use of carbonate contaminated deionized water.

	Z	AR IABI	ES			<u>LEVELS</u>
1.	As	shown	for	previous	experiments	1.80
2.	11	"	**	77	11	0.5 gms/liter
3.	11	11	"	11	11	128 to 135°F
4.	11	**	11	11	11	15 Minutes
5.	11	11	11	**	II	0 Inches
6.	11	11	11	**	Ħ	10 Minutes
7.	11	"	11	11	11	3 Cycles
8.	"	11	Ħ	11	II	52 to 54°F
9.	"	**	11	"	11	4.5
10.	"	11	**	"	11	NaOH
11.	11	**	11	II.	tī	1.30
12.	11	11	11	11	11	92 to 110°F
13.	11	"	**	11	11	0.009 N
14.	**	11	"	11	11	0.16 to 0.20 N
15.	11	11	"	"	11	7.8 to 8.7 N
16.	"	**	11	11	11	0.4 amps/in <sup>2</sup>
17.	11	11	11	11	11	15 Minutes
18.	11	11	11	11	11	See Appendix
19.	11	11	11	"	11	3 Cycles
20.	11	**	"	11	11	3 Cycles
21.	"	**	11	11	11	3 Types

### APPENDIX E

INDIVIDUAL PLAQUE RESPONSE MEASUREMENTS

#### POSITIVE RESPONSES (EXPERIMENT 1)

#### 1. Pickup Weight

#### LOT 151

PLAQUE AFTER I/P	AFTER FORMATION	LOSS
1. 64.4 gms	59.9	<b>-4.</b> 5
2. 63.3	58.1	<b>-</b> 5.2
3. 65.1	55.1	-10.0
LOT 152		
1. 51.9 gms	43.4	<b>-8.</b> 5
2. 50.6	43.3	<b>-7.</b> 3
3. 47.4	41.6	<b>-</b> 5.8
LOT 153		
1. 55.0 gms	50.5	<b>-</b> 4.5
2. 57.5	51.0	<b>-</b> 6,5
3. 59.0	53.5	<del>-</del> 5.5

#### 2. Capacity

Two terms are used to express electrical capacity: The first term is ampere-hours/plate which is the capacity of a single standard (approximately 15 in<sup>2</sup>) plate used throughout this work. The second term is ampere-hours/gram which may be considered an efficiency factor. The actual grams of active material impregnated in a test plate is determined and the ampere-hours capacity is expressed in terms of this quantity.

LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	2.42	0.228
2	4	2.40	0.226
3	2	2.42	0.228
LOT 152			
1	2	1.98	0.213
2	2	1.92	0.218
3	2	1.83	0.229
LOT 153			
1	2	2.07	0.284
2	4	1.98	0.305
3	2	2.10	0.276

Lot 153 impregnated plaques exhibited an unusually high efficiency factor in relation to the other lots in this experiment. No explanation can be given at this time to satisfactorily explain this peculiar result.

#### 3. Plaque Characterization

All three lots of plaques in this experiment exhibited a very rough surface texture. Active material appeared to be caked on the outside surface of the plaque. This observation is supported by the plaque weight loss associated with the formation process which to a certain extent affects the removal of an unimpregnated surface layer.

## POSITIVE RESPONSES (EXPERIMENT 2)

## 1. Pickup Weight

## LOT 151

PLAQUE AFTER I/P	AFTER FORMATION	LOSS
1. 42.2 gms	41.5	-0.7
2. 42.0	41.5	-0.5
3. 41.0	40.0	-1.0
LOT 152		
1. 47.2 gms	44.2	-3.0
2. 44.8	42.3	-2.5
3. 46.3	43.3	-3.0
LOT 153		
1. 42.6 gms	42.6	0.0
2. 41.1	40.6	<b>-</b> 0.5
3. 41.8	41.8	0.0

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	1.44	0.218
2	4	1.38	0.216
3	2	1.41	0.216
LOT 153			
1	2	1.44	0.218
2	4	1.40	0.209
3	2	1.29	0.239

#### 3. Plaque Characterization

The surface texture of the three groups of plaques in this experiment was much smoother than the previous experiment, but all surfaces were badly cracked. Lot 152 plaques were in such poor condition that they were not subjected to the capacity test.

#### POSITIVE RESPONSES (EXPERIMENT 3)

#### 1. Pickup Weight

#### LOT 151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	47.5	43.5	-4.0
2	51.0	47.0	-4.0
3	49.5	47.5	-2.0

#### 2. Capacity

#### LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	1.78	0.234

Test equipment failure resulted in the loss of the capacity data for plaques 2 and 3.

#### 3. Plaque Characterization

The results of this experiment were very poor. The surface of all plaques appeared to be blistered. In the case of Lots 152 and 153, the sintered nickel was removed from the body of the plaque during the wash procedure following the formation operation. This prevented the evaluation of pickup weight and capacity for these two lots.

### POSITIVE RESPONSES (EXPERIMENT 4)

As in the previous experiment, the surfaces of all plaques were blistered, but to a much greater extent. The condition of the plaque was so poor that they were not subjected to the formation operation and the experiment was discontinued.

## POSITIVE RESPONSES (EXPERIMENT 5A)

## 1. Pickup Weight

LOT	151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	54.5	54.0	-0.5
2	58.0	57.5	-0.5
3	56.0	56.0	0.0
LOT 152			
1	70.5	66.5	<b>-</b> 4.0
2	68.5	65.5	-3.0
3	64.5	56.5	-8.0
LOT 153			
1	54.5	54.0	<b>-0.</b> 5
2	60.0	59.5	-0.5
3	60.0	59.0	-1.0

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	2.37	0.263
2	4	2.59	0.254
3	2	2.37	0.279
LOT 152			
1	2	3.23	0.243
2	4	2.95	0.268
3	2	2.97	0.260
LOT_153			
1	2	2.16	0.251
2	4	2.54	0.226
3	2	2.54 E-8	0.249

### 3. Plaque Characterization

Lots 151 and 153 were very good. The plaques exhibited smooth clean surfaces without cracks. There was some material flaking associated with Lot 152 but no blistering.

#### POSITIVE RESPONSES (EXPERIMENT 6A)

### 1. Pickup Weight

### LOT 151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	*	57.5	
2	*	58.0	
3	*	65.0	
LOT 153			
1	*	60.0	
2	*	60.5	
3	*	64.5	

<sup>\*</sup>The pickup weights of the plaques after completion of the impregnation/polarization process were inadvertently missed during this experiment.

### 2. Capacity

### LOT 151

PLAQUE	<u>PLATE</u>	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	3.00	0.289
2	4	3.00	0.297
3	2	3.23	0.296
LOT 153			
1	2	3.00	0.286
2	4	2.88	0.280
3	2	3.15	0.281

### 3. Plaque Characterization

Lot 151 was good with fairly smooth and clean surfaces. Lot 152 was poor with active material flaking off. This lot was not weighed or subjected to the capacity test. Lot 153 was also poor with rough pitted surfaces.

#### POSITIVE RESPONSES (EXPERIMENT 7A)

This experiment was discontinued after completion of the third impregnation/polarization cycle because of poor plaque condition.

All three groups of plaques were badly blistered and exhibited flaking material.

### POSITIVE RESPONSES (EXPERIMENT 8B)

## 1. Pickup Weight

LOT :	151
LUT .	TOT

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	58.0	56.0	-2.0
2	59.5	57.0	-2.5
3	57.5	56.5	-1.0
LOT 152			
1	56.0	48.5	<b>-7.5</b>
2	59.5	52.0	<b>-7.</b> 5
3	61.5	54.5	<b>-7.</b> 0
LOT 153			
1	57.5	56.5	-1.0
2	57.5	57.0	-0.5
3	56.5	55.5	-1.0

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	2.41	0.257
2	4	2.47	0.257
3	2	2.36	0.268
LOT 152			
1	2	2.05	0.256
2	2	2.28	0.235
3	2	2.43	0.250
LOT 153			
1	2	2.17	0.236
2	4	2.32	0.242
3	2	2.12 E-13	0.252

### 3. Plaque Characterization

Lot 151 was very good with smooth and clean surfaces. Lot 152 was poor with material flaking off. Lot 153 was good, but exhibited a few cracks.

#### POSITIVE RESPONSES (EXPERIMENT 9A)

### 1. Pickup Weight

#### LOT 151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	69.0	67.0	-2.0
2	67.5	65.5	-2.0
3	68.0	67.0	-1.0
LOT 153			
1	72.0	68.0	-4.0
2	73.0	66.0	<b>-7.</b> 0
3	71.0	69.5	-1.5

### 2. Capacity

#### LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	4	2.82	0.274
2	2	2.97	0.275
3	4	2.94	0.272

#### 3. Plaque Characterization

Lot 151 was fair, but had a rough surface texture. Lot 152 was again very poor with flaking material. Lot 153 was also poor with a rough surface and cracks. Lots 152 and 153 were not subjected to the capacity test.

#### POSITIVE RESPONSES (EXPERIMENT 10B)

#### 1. Pickup Weight

## LOT 151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	47.5	46.5	-1.0
2	48.0	46.5	-1.5
3	48.0	47.5	<b>-</b> 0.5
<u>LOT 153</u>			
1	45.0	45.5	0.5
2	47.0	46.0	-1.0
3	44.5	43.5	-1.0

#### 2. Capacity

#### LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	1.93	0.254
2	4	2.02	0.250
3	2	1.85	0.243
LOT 153			
1	2	1.70	0.233
2	4	1.65	0.226
3	2	1.53	0.235

#### 3. Plaque Characterization

Lot 151 was good with a smooth and clean surface texture. Lot 152 was as usual, poor with flaking material. Lot 153 was fair, but the plaque surfaces contained a lot of cracks.

## NEGATIVE RESPONSES (EXPERIMENT 1)

## 1. Pickup Weight

LO	Т	15	1

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	49.2	47.2	-2.0
2	49.3	47.8	-1.5
3	45.9	43.9	-2.0
LOT 152			
1	55.9	54.9	-1.0
2	57.3	55.3	-2.0
3	53.9	51.4	<b>-2.</b> 5
LOT 153			
1	49.0	48.0	-1.0
2	50.6	49.1	<b>-1.</b> 5
3	48.6	46.6	-2.0

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAMS
1	2	1.98	0.268
2	4	2.08	0.274
3	2	1.84	0.267
LOT 152			
1	2	2.42	0.260
2	4	2.39	0.254
3	2	2.17	0.244
LOT 153			
1	2	2.12	0.279
2	4	2.15	0.262
3	2	1.89	0.255
		E-17	

### 3. Plaque Characterization

All three groups of plaques looked very good in this experiment. There was some cracking at the edges of the plaques associated with Lot 152.

## NEGATIVE RESPONSES (EXPERIMENT 2)

## 1. Pickup Weight

LOT	151

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	69.1	64.1	<b>-</b> 5.0
2	67.5	63.0	<del>-</del> 4.5
3	66.2	62.2	<del>-</del> 4.0
LOT 152			
1	78.5	73.0	<b>-</b> 5.5
2	80.6	75.1	<del>-</del> 5.5
3	79.7	73.7	-6.0
<u>LOT 153</u>			
1	67.0	62.0	<b>-</b> 5.0
2	67.0	63.0	-4.0
3	69.0	64.5	<b>-4.</b> 5

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	2.71	0.244
2	4	2.63	0.248
3	2	2.56	0.248
LOT 152			
1	2	3.00	0.240
2	4	3.12	0.238
3	2	3.18	0.221
LOT 153			
1	2	2.64	0.266
2	4	2.90	0.244
3	2	2.85 E-19	0.261

## 3. Plaque Characterization

The three groups of plaques in this experiment also looked very good.

Again, there was some cracking at the edges of the plaques associated with Lot 152.

## NEGATIVERESPONSES (EXPERIMENT 3)

# 1. Pickup Weight

LO	Т	1	5	1

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	69.5	65.0	-4.5
2	70.0	66.5	-3.5
3	71.0	67.0	<b>~4.</b> 0
LOT 152			
1	79.0	70.5	-8.5
2	78.5	70.0	-8.5
3	78.5	69.0	~9.5
LOT 153			
1	54.0	49.0	<del>-</del> 5.0
2	58.5	53.5	-5.0
3	<b>6</b> 6.0	61.5	-4.5

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	3.07	0.258
2	4	3.04	0.262
3	2	3.01	0.255
LOT 152			
1	3	3.48	0.268
2	4	3.09	0.258
3	1	3.29	0.259
LOT 153			
1	2	1.98	0.257
2	4	2.37	0.282
3	2	2.57 E-21	0.270

#### 3. Plaque Characterization

Lot 151 exhibited a slightly rough surface texture. Lot 152 was for the first time during these negative experiments very poor. The blistering phenomenon so common with the positive plaques occurred on the surface of this lot of plaques which resulted in material flaking off. Lot 153 was good with smooth clean surfaces.

## NEGATIVE RESPONSES (EXPERIMENT 4)

## 1. Pickup Weight

LOT	151	

PLAQUE	AFTER I/P	AFTER FORMATION	LOSS
1	41.0	38.0	-3.0
2	55.0	52.0	-3.0
3	54.0	51.5	-2.5
LOT 152			
1	66.0	63.0	-3.0
2	67.5	62.0	<b>-</b> 5.5
3	60.0	56.5	<b>-</b> 3.5
LOT 153			
1	52.5	50.5	-2.5
2	40.0	39.0	-1.0
3	41.5	39.5	-2.0

## 2. Capacity

## LOT 151

PLAQUE	PLATE	AMP-HOURS/PLATE	AMP-HOURS/GRAM
1	2	1.61	0.259
2	4	2.65	0.276
3	2	2.63	0.289
LOT 152			
1	2	2.93	0.279
2	2	2.92	0.278
3	4	2.62	0.276
LOT 153			
1	2	2.30	0.291
2	4	1.84	0.279
3	2	1.85 E-23	0.308

#### 3. Plaque Characterization

Lot 151 was good, but it exhibited a slightly rough surface texture.

Lot 152 was fair, but it exhibited a rough surface. Lot 153 was good with a smooth and clean surface.